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Note

## The Preparation of Sodium Perchlorate Labelled with Chlorine-36

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In order to investigate the effect of perchlorate ion on the thyroid gland<sup>1</sup> it was necessary to prepare <sup>36</sup>Cl labelled sodium perchlorate. The solution of <sup>36</sup>Cl in the form of 4.5 ml. 2.1 N hydrochloric acid, with specific activity ca. 120  $\mu\text{C/g}$  Cl obtained from Amersham (Code CIS. 1) was available as the source of radiochlorine. With regard to the experimental results of Izgaryshev *et al.*<sup>2</sup> radioactive perchlorate was prepared by anodic oxydation of  $\text{Ca}^{36}\text{Cl}_2$ .

This note reports the isolation of a  $\text{Na}^{36}\text{ClO}_4$  sample formed after electrolysis. The data concerning the dependence of the yield of perchlorate obtained by anodic oxidation on the concentration of the initial calcium chloride solution are also given.

The  $\text{Ca}^{36}\text{Cl}_2$  solution was prepared by neutralization of active hydrochloric acid with the slurry of calcium carbonate, the excess of which was filtered off.

The electrolysis cell consisted of a 15 ml. vessel with a thermostatic mantle. The vessel was provided with attachments for two Pt-electrodes of 4 sq. cm. each and an outlet for radioactive gaseous products which might evolve during electrolysis. A stirring electrode was trapped with mercury.

The anodic oxidation was carried out in a 5 ml. calcium chloride solution of various concentrations at the electric current 100 mA. During electrolysis the cell was cooled with tap water. The evolving of oxygen in bubbles which occurred after ca. 40–80 hrs, indicated the end of the reaction<sup>2</sup>.

After electrolysis, the content of the cell was transferred to the beaker, an excess of sodium carbonate solution was added, calcium carbonate precipitated was filtered off and the solution of sodium salts (perchlorate) evaporated to dryness. The cold residue was extracted six times with 10 ml. portions of anhydrous ethyl acetate<sup>3</sup>. The organic extract was filtered and the solvent evaporated. The samples of sodium perchlorate were finally dried at 110°C and weighed for the chemical yield.

Owing to the possible presence of chlorine in the lower oxidation states, the chemical purity of sodium perchlorate was determined by precipitating chloride and chlorate after reduction with silver nitrate. The amounts of silver chloride were expressed as sodium chloride (Table I).

The results in Table I indicate that a still satisfactory perchlorate yield (Exps. 3 and 4) could be obtained from calcium chloride solution of the same concentration as that in the original  $\text{H}^{36}\text{Cl}$ . However, the specific activity of the radioactive sample obtained in the yield of more than 90 per cent was somewhat lowered to ca. 70  $\mu\text{C/g}$ . Cl owing to the addition of some inactive calcium chloride in the course of the preparation of the primary chloride solution from the original 2.1 N  $\text{H}^{36}\text{Cl}$ .

TABLE I

Exp No.	CaCl <sub>2</sub> conc.	Yield of perchlorate %	Impurity, expressed as NaCl, %
1	0.42 N	59.6	0.6
2		54.1	0.4
3	2.10 N	77.2	0.0
4		79.4	0.0
5	3.60 N	83.2	0.5
6		94.7*	less than 0.5*

\* Figures show the results of the experiment with radiochlorine.

While this note was being prepared for print the results of Billigmeier and McDonald<sup>4</sup> concerning the preparation of labelled potassium perchlorate were published. Starting from a 0.926 N lithium chloride solution the authors succeeded in obtaining a perchlorate yield of more than 90 per cent.

## REFERENCES

1. I. Šimonović, *Thesis*, Zagreb 1963.
2. N. A. Izgarišev and S. V. Gorbačev, *Kurs teoretičke elektrohimii*, Gosshimizdat, Moskva, 1951, p. 441.
3. A. I. Vogel, *Quantitative Inorganic Analysis*, Longmans, Green and Co., London 1957, p. 497.
4. J. E. Billigmeier and R. L. McDonald, *J. Inorg. Nucl. Chem.* **26** (1964) 663.

## IZVOD

## Priprava natrijeva perklorata označenoga klorom-36

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Na<sup>36</sup>ClO<sub>4</sub> pripravljen je metodom anodne oksidacije Izgariševa i dr. Prikažana je izolacija uzorka Na<sup>36</sup>ClO<sub>4</sub> nakon elektrolize otopine Ca<sup>36</sup>Cl<sub>2</sub>. Dani su podaci o ovisnosti iskorištenja dobivenog perklorata o koncentraciji početne otopine kalcijeva klorida.

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Preliminary Note

## Untersuchungen im System $N_2H_6F_2$ - $UF_6$ -HF

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In einer Fluorothenepruvette wurde zu einer erfrorzten Lösung von Hydrazinfluorid in wasserfreiem Fluorwasserstoff Uranhexafluorid im Ueberschuss kondensiert. Die enttaute Lösung ist klar und anfänglich hellgelb. Bei weiterem Stehen wird die Lösung gelborange und schliesslich scheiden sich gelbe Kristalle aus. Nach 24 Stunden wurden der überschüssige Fluorwasserstoff und das Uranhexafluorid abgesaugt und das Reaktionsprodukt noch längere Zeit im Hochvakuum bei Zimmertemperatur getrocknet. Das Reaktionsprodukt ist ein gelbes lockeres Pulver. Das Produkt wurde unter Argon in Glasampullen zur Analyse verteilt und abgeschmolzen. Die Zusammensetzung des Produktes entspricht der Formel  $N_2H_5UF_7$ :

Anal. Ber.:  $N_2H_4$  7,92; U 58,91; F 32,92%

Gef.:  $N_2H_4$  8,6; U 59,6; F 32,3%

Weitere Untersuchungen sind im Gange.

### IZVLEČEK

#### Raziskovanja v sistemu $N_2H_6F_2$ — $UF_6$ -HF

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Opisana je sinteza ternarnega uranovega fluorida v brezvodnem fluorovodiku. Sestava produkta odgovarja formuli  $N_2H_5UF_7$ .

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