EUR 3656 e

EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

THE PRECISE AND ACCURATE DETERMINATION OF LITHIUM HYDROXIDE SOLUTIONS

by

J. PAUWELS, Y. LE DUIGOU and K.F. LAUER

1967

Joint Nuclear Research Center
Geel Establishment - Belgium

Central Bureau for Nuclear Measurements - CBNM

LEGAL NOTICE

This report is on sale at the addresses listed on cover page 4

at the price of FF 4.–	FB 40.–	DM 3.20	Lit. 500	Fl. 3.–

When ordering, please quote the EUR number and the title, which are indicated on the cover of each report.

Printed by SMEETS
Brussels, December 1967

EUR 3656 e

THE PRECISE AND ACCURATE DETERMINATION OF LITHIUM HYDROXIDE SOLUTIONS

by J. PAUWELS, Y. LE DUIGOU and K.F. LAUER

European Atomic Energy Community - EURATOM
Joint Nuclear Research Center - Geel Establishment (Belgium)
Central Bureau for Nuclear Measurements - CBNM

Brussels, December 1967 - 26 Pages - 2 Figures - FB 40

Four methods for the precise determination of lithium hydroxide solutions have been compared : the acid-base titration with hydrochloric acid, the acid-base titration with benzoic acid, the weighing as sulphate and the weighing as carbonate.

For the determination of a 0.1 M solution the agreement between the four results was better than 0.02 %.

EUR 3656 e

THE PRECISE AND ACCURATE DETERMINATION OF LITHIUM HYDROXIDE SOLUTIONS

by J. PAUWELS, Y. LE DUIGOU and K.F. LAUER

European Atomic Energy Community - EURATOM
Joint Nuclear Research Center - Geel Establishment (Belgium)
Central Bureau for Nuclear Measurements - CBNM
Brussels, December 1967 - 26 Pages - 2 Figures - FB 40

Four methods for the precise determination of lithium hydroxide solutions have been compared : the acid-base titration with hydrochloric acid, the acid-base titration with benzoic acid, the weighing as sulphate and the weighing as carbonate.

For the determination of a 0.1 M solution the agreement between the four results was better than 0.02 %.

EUR 3656 e

EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

THE PRECISE AND ACCURATE DETERMINATION OF LITHIUM HYDROXIDE SOLUTIONS

by

J. PAUWELS, Y. LE DUIGOU and K.F. LAUER

1967

Joint Nuclear Research Center
Geel Establishment - Belgium

Central Bureau for Nuclear Measurements - CBNM

SUMMARY

Four methods for the precise determination of lithium hydroxide solutions have been compared : the acid-base titration with hydrochloric acid, the acid-base titration with benzoic acid, the weighing as sulphate and the weighing as carbonate.

For the determination of a 0.1 M solution the agreement between the four results was better than 0.02 %.

KEYWORDS

DETERMINATION
LITHIUM HYDROXIDES
SOLUTIONS
TITRATION
HYDROCHLORIC ACID
BENZOIC ACID
GRAVIMETRY
LITHIUM SULPHATES
LITHIUM CARBONATES

Contents

1. INTRODUCTION

The purpose of this study is the development of a method for determination of lithium with an accuracy better than 0.05 %.
It can be seen from the literature survey, that lithium is normally determined by gravimetric or volumetric methods. The obtained precisions were generally a factor 10 to 100 too high for our purpose.

By gravimetry, lithium was determined as sulphate [1,2,3], phosphate [4,5,6,7], nickel- [8] and zinc- uranyl-acetate [9,10], arsenate [11], periodate [12,13], and aluminiumperiodate [14]. For determinations of a few milligrammes of lithium, the precisions were of the order of 0.1 to 1 %.

The volumetric methods are mostly indirect and are used after isolation of the lithium.

After extraction of lithium chloride with 2-ethyl 1-hexanol, the alcoholic phase can be titrated potentiometrically with silver nitrate with a precision of 0.16 % [15] or according to the Volhard method [16]. Lithium sulphate solutions were titrated conductometrically with barium hydroxide [17].

After precipitation of lithium as periodate, a titration with standardized thiosulphate or arsenic is possible [18].

After a separation of heavy metals [19] and alkaline earth metals [20] on ion exchange resins the total alkalis were titrated with precisions of 0.2 % with hydrochloric acid. Lithium salt solutions were also analysed by titration of H^+ liberated after complexation with EDTA [21].

Traces of lithium are usually determined by colorimetry, using thoron [22,23,24] and quinazolinazo [25] as reagents. By spectrophotometric titration with 1.3- dimethyl-violuric acid precisions of 0.3 to 1.5 % were obtained [26].

Manuscript received on July 11, 1967.

Finally traces were also determined by polarography [27,28,29] and flame photometry [30].

As a conclusion the methods which seemed us to be the most promising, provided that precise techniques are used and are applied to relatively pure solutions, were the acid-base titration of lithium hydroxide and gravimetry as sulphate or carbonate.

A study of four methods - titration with hydrochloric acid, titration with benzoic acid, weighing as sulphate and weighing as carbonate - will give indications of the accuracy that can be expected for the chemical definition of a relatively pure lithium hydroxide solution.

2. PRINCIPLE OF THE METHODS

The accurate determination of lithium hydroxide will be based on the combination of these four methods.

The titration with hydrochloric acid is done potentiometrically, using a combined glass- saturated calomel electrode and a recording pH- meter for the end point localisation. Lithium hydroxide and hydrochloric acid (about 20g of 0.1 M solutions) are weighed to 0.1 mg in weighing burets. The precise addition of reagents for the end point localisation of the titration is made with a calibrated microburet.

The titration with benzoic acid is also done potentiometrically, using the same equipment. The end point is precisely determined by back titration of an excess of added lithium hydroxide.

The two gravimetric methods are based on the fact that lithium sulphate and lithium carbonate are very stable and non hygroscopic salts.

According to C. Duval [2] the thermogravimetric curve of lithium sulphate shows a horizontal section between 200 and 877°C whereas lithium carbonate is stable up to 428°C, so that they can be easily dried.

Measurements were made of air temperature, barometric pressure and humidity during all weighings and the appropriate corrections for air buoyancy were applied.

3. EXPERIMENTAL DETAILS OF METHODS

3.1. The acid-base titration with hydrochloric acid

3.1.1. Method of determination

15 minutes before starting the titration, nitrogen (purity 99.999995 %) is passed through the empty titration cell in order to eliminate all carbon dioxide.

A weighed quantity of about 20 g 0.1 M hydrochloric acid is introduced into the titration cell and lithium hydroxide is added dropwise from a weighing buret. Thus during the major part of the titration time one has a large excess of hydrochloric acid in the cell and so the greater part of possibly present lithium carbonate is decomposed before the real end point determination begins. Lithium hydroxide is added until the pH reaches a constant value between 8 and 9 (excess of LiOH). The added quantity is also weighed to 0.1 mg.

Then, with a calibrated microburet, 2 to 5 droplets of 0.1 M hydrochloric acid are added, until the equivalence point is reached. The weight of one droplet was experimentally determined as being 4.80 mg ± 0.15 mg. After each addition of an acid droplet, one must wait about 20 minutes until the pH value is constant. To determine precisely the equivalence point of the titration one can interpolate by means of the following formula [31]:

$$V_f = V_2 + \frac{(h_2 - h_1)}{(h_2 - h_1) + (h_2 - h_3)} \cdot s$$

V_f : weight of hydrochloric acid at the end point of the titration

V_2 : weight of hydrochloric acid just one droplet before it.

h_2, h_1, h_3 : differences in potential (in mV), resp. at the maximal potential jump, before and after it.

s : weight of one hydrochloric acid droplet.

3.1.2. Standardization of 0.1 M hydrochloric acid

0.1 M hydrochloric acid was prepared by dilution of HCl (d = 1.19 - analytical grade) with bidistilled water and standardized gravimetrically as silver chloride. Since photolytical decomposition of silver chloride can give errors reaching - 0.2 % [1], the precipitation was sheltered from light. Precipitations were done on approximately 0.5 g silver, since L. Erdey indicates that for such amounts the most accurate results are obtained [1].

3.1.2.1. The silver chloride method for the determination of silver

About 0.5 g of silver (purity: 99.99 %) is dissolved in 5 ml 2M nitric acid and diluted to 125 ml with bidistilled water. After heating to approximately 70°C, one adds a slight excess (20 - 25 ml) of 0.2 M hydrochloric acid dropwise from a pipette, while stirring constantly. After storing for one night in a dark place, the solution is decanted through a glass filter crucible of porosity 3 to 4, the content of the beaker is 4 times washed with 12.5 ml 0.1 M nitric acid and then each time decanted again. The precipitate is transferred quantitatively to the filter crucible and again washed with approximately 50 ml 0.01 M nitric acid.

After drying for 30 minutes at 100°C and then for 1 hour at 130 - 150°C, the precipitate is weighed.

3.1.2.2. The silver chloride method for the determination of hydrochloric acid

5 ml 2 M nitric acid is added to about 40 ml of 0.1 M hydrochloric acid and diluted to 125 ml with bidistilled water.

After heating to approximately 70°C, one adds a slight excess (20 ml) of 0.25 N silver nitrate solution dropwise from a pipette, while stirring constantly.
After storing for one night in a dark place, the precipitate is washed, filtered and dried as described above.

3.1.2.3. Solubility effects

At 20°C 1 l of pure water dissolves 1.43 mg of silver chloride. Since for the described silver- and hydrochloric acid determination methods 250 ml of solution are needed, the amount of dissolved silver chloride could be 0.36 mg and this would give a systematic deviation of - 0.05 to - 0.07 %. An experimental difference corresponding to this value was found.
However the following considerations make it likely that this difference is more due to photolysis than to solubility effects:

- In each experiment there was an excess of Ag^+ or Cl^- ions, which reduce the dissolved quantity of silver chloride (1 liter 1 % hydrochloric acid dissolves 0.2 mg of silver chloride [1]).
- It is not sure that during the washing step the precipitate reaches equilibrium with the solution.

- All solutions were acidified with nitric acid to avoid the formation of colloidal silver chloride. Values about the solubility of silver chloride in nitric acid could not be found.
- Finally it seems that silver chloride precipitates can contain 0.02 % of water, which is only removed at approximately its melting point (445°C) [1].

3.1.2.4. Photolytic effects

A series of silver chloride precipitates were weighed after 1 and 5 hours exposure to artificial light. After 5 hours constancy was reached. The difference with the theoretical quantity of precipitate was - 0.15 %.

3.1.2.5. Conclusion

Since it seems very difficult to estimate precisely the corrections for solubility and photolytic effects, it was prefered to use Ag as a primary standard and to introduce an experimental correction of - 0.07 % on the AgCl determination.
The hydrochloric acid determinations were always run simultaneously with silver determinations, assuming that a determination of chloride with silver gives rise to the same systematic error as a silver determination with chloride.

3.2. The acid-base titration with benzoic acid

About 0.35 g 99.98 % pure benzoic acid (NBS- titrimetric standard N° 350) is weighed to 0.01 mg and dissolved in 20 ml absolute ethanol. After removing carbon dioxide from the titration cell, one neutralizes the benzoic acid with a weighed quantity of 0.1 M lithium hydroxide, until the pH reaches a constant value between 9.5 and 10.

Then, the end point is precisely determined by back titration with 0.1 M hydrochloric acid as described in 3.1.
As the amount of hydrochloric acid added is only about 0.1 eq. % of the amount of benzoic acid, the possible systematic error of the back titration with hydrochloric acid instead of benzoic acid is practically negligible.

The molarity of the lithium hydroxide solution is given by:

$$M_{LiOH} = \frac{mg_{B.A.}}{122.12 \times g_{LiOH}} + \frac{g_{HCl} \times M_{HCl}}{g_{LiOH}}$$

M_{LiOH} : molarity of lithium hydroxide

M_{HCl} : molarity of hydrochloric acid

$mg_{B.A.}$: weight of benzoic acid (mg)

g_{HCl} : weight of hydrochloric acid solution (gram)

g_{LiOH} : weight of lithium hydroxide solution (gram)

It is assumed that the density of the solution is exactly 1 in the formula used for molarity.

3.3. The lithium sulphate method

About 20 g of 0.1 M lithium hydroxide is weighed to 0.1 mg and quantitatively transferred to a platinum crucible. After addition of a calculated excess of concentrated sulphuric acid (about 0.2 ml) the solution is slowly evaporated and dried at 750°C until a constant weight is obtained.

3.4. The lithium carbonate method

65 to 75 g of a 0.1 lithium hydroxide solution is weighed to 0.1 mg and quantitatively transferred to a 200 ml teflon beaker.

The hydroxide is then transformed to carbonate by passing during 1 - 2 hr a flow of carbon dioxide through the solution.

The solution is then quantitatively transferred to a platinum crucible, evaporated carefully and dried at 350 - 375°C until constant weight is reached.

4. SYSTEMATIC ERRORS

4.1. Influence of carbonate in the lithium solution on the acid base titration with hydrochloric acid

When lithium hydroxide is in contact with the atmosphere it absorbs carbon dioxide.
Since in the titration described in 3.1. all the hydroxide is titrated first and then the carbonate, the titration curve has an irregular shape in the vicinity of the end point (Fig. 1).

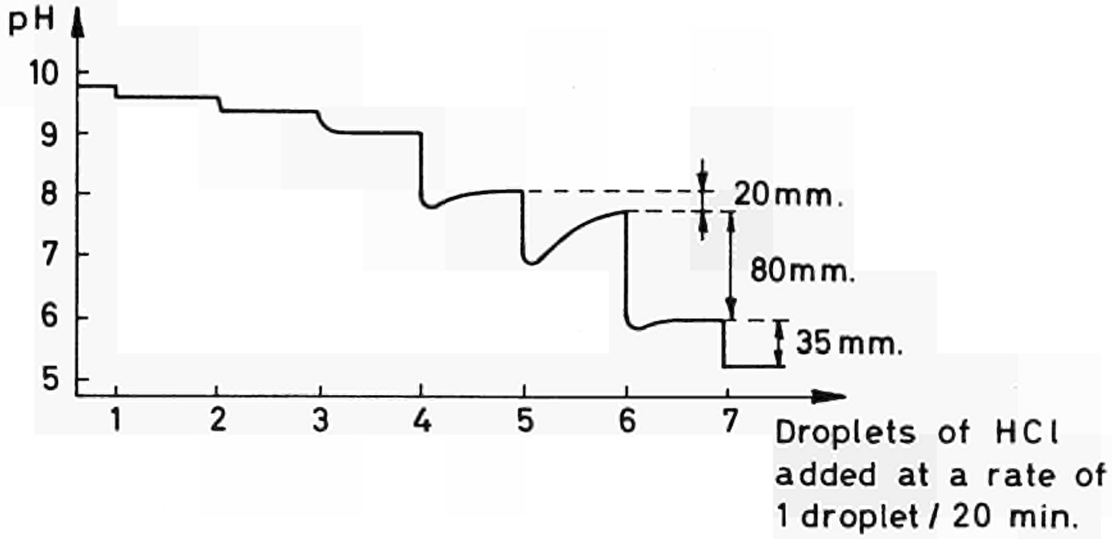
At the beginning of the back titration of lithium hydroxide, the pH reaches immediately a constant value after the addition of each drop of acid.

After the end of the titration of the hydroxide and carbonate, a series of curved parts appears in the titration curve, which indicate the transformation of lithium bicarbonate into chloride and the slow removal of carbon dioxide from the solution with the nitrogen flowing over the solution.

Based on this remark one can determine the number of acid drops which are needed to titrate the bicarbonate in the solution. The acid needed for this transformation can be calculated with a precision of 10 - 20 % (Fig. 2).
By exposing aliquots of the same lithium hydroxide solution during different times to atmosphere, solutions with different carbonate concentrations were prepared and titrated. Table I shows that the apparent lithium content must be corrected by 0.01 % for a carbonate content of 0.2 eq. %.

4.2. Influence of total anions in the lithium solution

For the two acid-base titrations, the anionic impurities other than carbonate should be smaller than 0.01 eq. %.



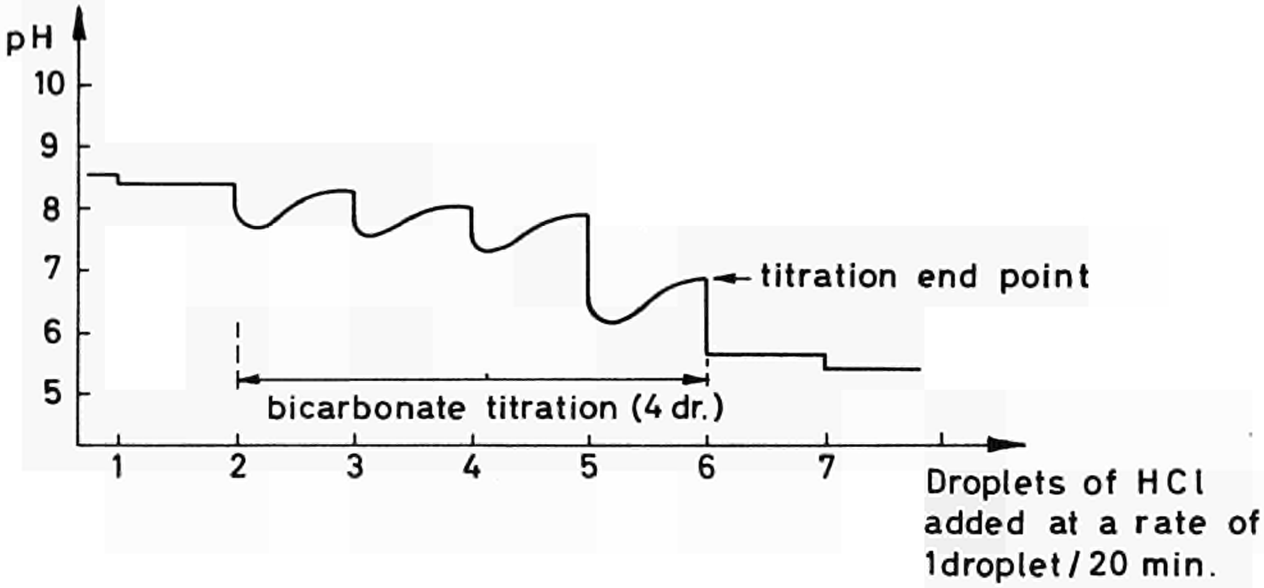
— Weighed amount of added HCl : 21.0471 g.

— Weighed amount of added LiOH : 19.3902 g.

— HCl added with microburet : $5 \times 4.8\ \text{mg} + \frac{60}{60+45} \cdot 4.8\ \text{mg} = 26.9\ \text{mg}$

— Molarity of LiOH: $\frac{0.09719 \times (21.0471 + 0.0269)}{19.3902} = 0.10563\ \text{M}$.

Fig 1. Titration curve of LiOH with 0.1 M HCl.
(determination number 3 of Table III)



Example:

- Total amount of HCl : 19 200 mg
- Amount of HCl needed for bicarbonate titration : 4.8 mg x 4 = 19.2 mg
- Equivalent % of bicarbonate : $\frac{19.2}{19\,200} \times 100 = 0.1$ eq.%

Fig 2. Determination of bicarbonate content of LiOH by titration of LiOH with HCl 0.1 M

The titration with hydrochloric acid can be corrected for carbonate concentrations ranging from 0.2 to 0.6 eq. %. (cf. point 4.1.)
Such carbonate concentrations do not have a perceptible influence on the benzoic acid titration in alchoholic medium. The detailed study of this phenomenon is still going on. The actual results were corrected in the same way as for the hydrochloric acid titration. Anyway, this correction has only an influence of 0.008 % on the value of the benzoic acid titration and of 0.002 % on the mean value of the four methods.

In the sulphate method, volatile anions (Cl^-, CO_3^{--}, etc.) do not interfere. In the carbonate method they do, just as the non volatile ones. (see Table II).

4.3. Influence of cationic impurities

The influence of alkali- and alkaline earth metals on the determination of lithium hydroxide by acid base titration will be expressed in equivalents. This means that an interference of 1 ppm (= 1 μg/gram lithium contained) has a significantly smaller influence on the lithium hydroxide determination (Table II).
From Table II it appears that the interference becomes more important when the interfering ion has a smaller atomic weight and a larger valency.
The influence of metal ions on the determination of lithium hydroxide as sulphate is given by:

$$\frac{M_{M_x(SO_4)_y}}{x\ A_M} \cdot \frac{2A_{Li}}{M_{Li_2SO_4}}$$

$M_{M_x(SO_4)_y}$: molecular weight of the metal sulphate

$M_{Li_2SO_4}$: molecular weight of the lithium sulphate

A_M : atomic weight of the metal

A_{Li} : atomic weight of lithium

It can be calculated, that for the sulphate method a similar effect exists as for the titration (Table II). The influence of metal ions on the determination of lithium hydroxide as carbonate is given by:

$$\frac{M_{M_x(CO_3)_y}}{x\ A_M} \times \frac{2A_{Li}}{M_{Li_2CO_3}}$$

$M_{M_x(CO_3)_y}$: molecular weight of the metal carbonate

$M_{Li_2CO_3}$: molecular weight of lithium carbonate

A_M : atomic weight of the metal

A_{Li} : atomic weight of lithium

The results of the calculations are also given in Table II.

5. RESULTS

Four silver determinations gave a yield of (99.93 ± 0.03)% and 4 simultaneous acid determinations a molarity of 0.09712 (s= 0.00005).
From this two values a corrected molarity of 0.09719 M ± 0.00005 M was calculated for HCl used.

The lithium hydroxide solution, whose carbonate content was determined as 0.16 eq. %, was analyzed by the four described methods. A comparison of the obtained results is given in Table III.

To obtain the accurate values one has to make several corrections for systematic errors. These corrections and their precisions are summarized in Table IV.
Disturbing impurities were determined by emission spectrography or taken from the certificate of analysis of the lithium used.

The carbonate content of the lithium hydroxide solution was determined experimentally as described in 4.1. (Fig. 2). For the benzoic acid titration, a blank value for acidic traces present in ethanol was determined. This was done by titration of 20 ml ethanol and 27 ml carbonate free bidistilled water (corresponding to the amount of lithium hydroxide solution used for a titration with benzoic acid) with 0.03 M lithium hydroxide.
As one can see in Tables III and IV the correction for systematic errors are smaller than the standard deviations of the different methods. A comparison of the final results is given in Table V. Their agreement is better than 0.02 %.

6. CONCLUSION

A comparison of the results of four methods - acid-base titration with hydrochloric acid, acid-base titration with benzoic acid, weighing as sulphate and weighing as carbonate - allows the determination of the molarity of 0.1 M lithium hydroxide solutions with an accuracy consistent with the uncertainty of the measurements.

ACKNOWLEDGEMENTS

The authors thank Prof. J. Hoste, Dr. J. Spaepen, Dr. G.H. Debus and Dr. P.J. De Bièvre for their encouragement and interest in this work.

J.A. Pauwels wishes to thank especially Prof. J. Hoste, under the direction of which he carries on a thesis, for his critical discussions.

REFERENCES

(1) ERDEY, L., Gravimetric analysis, Part II, 1st English Ed. Pergamon, London (1965)

(2) DUVAL, C., Traité de microanalyse minérale Tome I, 1st Ed. Presses Scientifiques Internationales (1954).

(3) WILSON, C.L. and WILSON, D.W., Comprehensive Analytical Chemistry, Vol. IC, 1st Ed., Elsevier, Amsterdam

(4) CALEY, E.R. and SIMONS, B.A., jr., Anal. Chem. 25 (1953) 1386.

(5) KINDYAKOV and KOKHLOVA, Trudy Moskov Tonkoi Khim. Tekhnol. im MV Lomonosova 6 (1956) 9

(6) VISHVESHWARAIAH, K.N. and PATEL, C.C., J. Indian Inst.Sci. 41A (1959) 16.

(7) KATO, T. and HAGIWARA, Z., Technol. Repts. Tohoku Univ. 14 (1950) 21.

(8) SHIGEMATSU and KIMURA, H., J. Chem. Soc. Japan, Pure Chem. Sect. 73 (1952) 443.

(9) KIMURA, H. and YASUMARU, M., J. Chem. Soc. Japan, Pure Chem. Sect. 73 (1952) 378.

(10) GRÜTTNER, B., Z. anal. Chem. 133 (1951) 36.

(11) GASPAR Y ARNAL, T., Chim. et Ind. (Paris) 83 (1960) 406.

(12) RESNIK, KISER and DALTON, Proc. Iowa Acad. Sci. 60 (1953) 315.

(13) BAGBANBY, I.L.and GUSENOV, I.K., Azerb. Khim. Zhur. 5 (1960) 77

(14) SOOS, I., VARHEYI, C. and SOMAY, M., Studia Univ. Barbes Bolaya, Ser. 1, n° 2 (1959) 71.

(15) WATERBURRY,G.R., VAN KOOTEN, E.H. and MOROSIN, B., Anal. Chem. 30 (1958) 1627.

(16) WHITE, J.C. and GOLDBERG, G., ORNL - 1827 (1955)

(17) HEMMELER, Ann. Chim. Appl 37 (1947) 119.

(18) ROGERS, L.B. and CALEY, E.R., Ind.eng. Chem. 15 (1943) 209

(19) SAMUELSON, O. and SCHRAMM, K., Z. Electrochem. 57 (1953) 207

(20) SAMUELSON, O. and SJÖSTRÖM, E., Anal. Chem. 26 (1954) 1908

(21) SCHWARZENBACH, G. and SCHNORF, P., Schw. P. 245126 (1945/47).

(22) NIKOLAEV, A.V. and SOROKINA, A.A., Dokl. Akad. Nauk. SSSR 77 (1951) 427.

(23) THOMASON, P.F., Anal. Chem. 28 (1956) 1527.

(24) SANDELL, E.B., Colorimetric determination of trace metals, 3rd Ed., N.Y. London (1959).

(25) DZIOMKO, V.M., ZELICHENSK, S.L. and MARKOVICH, I.S., Zhur. Anal. Khim. 18 (1963) 937.

(26) TAYLOR, M.E., and ROBINSON, R.J., Anal. Chem. 34 (1962) 533.

(27) ZLOTOWSKI, I. and KOLTHOFF, I.M., Ind. eng. Chem., anal. Edit. 14 (1942) 473.

(28) PANCHENKOW, G.M., KUZNETZOVA, E.M. and AKSHINSKAYA, N.V., Zhur. Anal. Khim. 15 (1960) 424.

(29) SCHÖBERG, G. and GUTTMANN, V., Mikrochim. Acta (1958) 319.

(30) CEA - ~~tr~~ - R - 1570 (1962).

(31) LAUER, K.F. and LE DUIGOU, Y., Z. anal. Chem. 184 (1961) 4.

Table I

Influence of the carbonate content on the determination of lithium by acid-base titration with hydrochloric acid.

Estimated bicarb. content	Apparent Li molarity determined	Standard deviation (s) n = 6
0.11 eq. %	0.10232	0.000015
0.20 eq. %	0.10231	0.000014
0.22 eq. %	0.10231	0.000020
0.31 eq. %	0.10230	0.000016

Table II

Systematic errors caused by impurities in the lithium hydroxide solution.

Element	Calculated effect of 1 μg impurity/g lithium (in 10^{-4} %)		
	Titrations	sulphate method	carbonate method
Na	+ 0.30	+ 0.38	+ 0.43
K	+ 0.18	+ 0.28	+ 0.34
Rb	+ 0.08	+ 0.19	+ 0.25
Cs	+ 0.05	+ 0.18	+ 0.22
Be	+ 1.56	+ 1.50	+ 1.45
Mg	+ 0.58	+ 0.64	+ 0.66
Ca	+ 0.34	+ 0.44	+ 0.47
Sr	+ 0.16	+ 0.27	+ 0.32
Ba	+ 0.10	+ 0.22	+ 0.26
Cl	+ 0.20	-	+ 0.15
Si	-	- 0.22	- 0.18

Table III

Results for the determination of one lithium hydroxide solution by the four described methods.

N°	EXPERIMENTAL DETERMINED MOLARITY OF LiOH			
	HCl - titration	Benz. Ac. titration	sulphate	carbonate
1	0.10560	0.10564	0.10562	0.10560
2	0.10561	0.10565	0.10567	0.10565
3	0.10563	0.10565	0.10561	0.10565
4	0.10560	0.10566	0.10567	0.10560
5	-	-	-	0.10563
6	-	-	-	0.10564
M	0.105610	0.105650	0.105642	0.105628
s	0.000014	0.000008	0.000032	0.000023

Table IV

Calculated corrections for systematic errors.

Method	Cause of systematic error	Magnitude of error (10^{-6} M)	Standard deviation of the determination of the error (10^{-6} M)
HCl-titration	0.16 eq. % carbonate	- 8.4	± 1.8
	30 ppm Na	+ 0.9	± 0.18
	45 ppm K	+ 0.8	± 0.16
	20 ppm Mg	+ 1.1	± 0.22
	10 ppm Ca	+ 0.3	± 0.06
	41 ppm Cl	- 0.8	± 0.16
	total systematic error	- 6.1	± 2.6
Benzoic acid titration	0.16 eq. % carbonate	- 8.4	± 1.8
	acidity of ethanol	-10.5	± 2.0
	titre of benzoic acid	+21.0	titrimetric standard
	30 ppm Na	+ 0.9	± 0.18
	45 ppm K	+ 0.8	± 0.16
	20 ppm Mg	+ 1.1	± 0.22
	10 ppm Ca	+ 0.3	± 0.06
	41 ppm Cl	- 0.8	± 0.16
	total systematic error	+ 4.4	± 4.6
Lithium sulphate	ash residue of H_2SO_4	< 1.0	± 0.5
	30 ppm Na	+ 1.1	± 0.22
	45 ppm K	+ 1.3	± 0.26
	20 ppm Mg	+ 1.3	± 0.26
	10 ppm Ca	+ 0.4	± 0.08
	15 ppm Si	- 0.3	± 0.06
	total systematic error	+ 4.3	± 1.4
Lithium carbonate	30 ppm Na	+ 1.3	± 0.26
	45 ppm K	+ 1.5	± 0.30
	20 ppm Mg	+ 1.3	± 0.26
	10 ppm Ca	+ 0.5	± 0.10
	15 ppm Si	+ 0.3	± 0.06
	41 ppm Cl	+ 0.6	± 0.12
	total systematic error	+ 5.5	± 1.1

Table V

Comparison of the results obtained with the four described methods.

Method	Uncorrected value			Corrected value
	M	s	s %	
1) HCl titration	0.105610	0.000014	0.013	0.105616
2) Benz. Ac. titration	0.105650	0.000008	0.008	0.105637
3) Lithium sulphate	0.105642	0.000032	0.030	0.105638
4) Lithium carbonate	0.105628	0.000023	0.022	0.105623
	M = 0.105632			M = 0.105629 s_M = 0.000011 s_M% = 0.010

NOTICE TO THE READER

All Euratom reports are announced, as and when they are issued, in the monthly periodical **EURATOM INFORMATION,** edited by the Centre for Information and Documentation (CID). For subscription (1 year : US$ 15, £ 5.7) or free specimen copies please write to :

Handelsblatt GmbH
"Euratom Information"
Postfach 1102
D-4 Düsseldorf (Germany)

or

Office central de vente des publications
des Communautés européennes
2, Place de Metz
Luxembourg

To disseminate knowledge is to disseminate prosperity — I mean general prosperity and not individual riches — and with prosperity disappears the greater part of the evil which is our heritage from darker times.

Alfred Nobel

SALES OFFICES

All Euratom reports are on sale at the offices listed below, at the prices given on the back of the front cover (when ordering, specify clearly the EUR number and the title of the report, which are shown on the front cover).

OFFICE CENTRAL DE VENTE DES PUBLICATIONS DES COMMUNAUTES EUROPEENNES
2, place de Metz, Luxembourg (Compte chèque postal N° 191-90)

BELGIQUE — BELGIË
MONITEUR BELGE
40-42, rue de Louvain - Bruxelles
BELGISCH STAATSBLAD
Leuvenseweg 40-42, - Brussel

DEUTSCHLAND
BUNDESANZEIGER
Postfach - Köln 1

FRANCE
SERVICE DE VENTE EN FRANCE
DES PUBLICATIONS DES
COMMUNAUTES EUROPEENNES
26, rue Desaix - Paris 15e

ITALIA
LIBRERIA DELLO STATO
Piazza G. Verdi, 10 - Roma

LUXEMBOURG
OFFICE CENTRAL DE VENTE
DES PUBLICATIONS DES
COMMUNAUTES EUROPEENNES
9, rue Goethe - Luxembourg

NEDERLAND
STAATSDRUKKERIJ
Christoffel Plantijnstraat - Den Haag

UNITED KINGDOM
H. M. STATIONERY OFFICE
P. O. Box 569 - London S.E.1

EURATOM — C.I.D.
51-53, rue Belliard
Bruxelles (Belgique)