Production of stable isotopes of light elements: past, present and future

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Abstract. Taking into account the world trends in the production and use of stable isotopes of light chemical elements, the main directions of their application and the scale of modern production are considered. Particular attention is paid to stable isotopes, mainly used in clinical medicine and various biological studies. At examples for isotopes of $^{13}$C, $^{18}$O and $^{15}$N are analyzed the change in the production volume of isotope products by years, the technologies used for isotope enrichment are considered and manufacturers in different countries of the world are indicated. Particular attention is paid to the appearance of relatively new industrial processes for the enrichment of $^{13}$C and $^{18}$O. The dominant role of physicochemical separation methods in the production of light isotopes is noted. In addition, from the technology development point of view, technical proposals for the improvement of physicochemical separation methods aimed at enriching $^{15}$N and separation of boron isotopes are considered.

1. Introduction

What nomenclature of stable isotopes of light elements is most in demand now and will be in demand in the future? Which method and what isotope separation processes will dominate in the coming years, and, accordingly, on what processes of isotope enrichment are to be bet on?

Answers to these questions can be obtained on the basis of an analysis of the history of development and the scale of isotopes production and, of course, the technologies used.

2. Main applications of stable isotopes of light elements

2.1. Stable isotopes of light elements

As a rule, stable isotopes of light elements include isotopes of hydrogen, helium, lithium, boron, carbon, nitrogen and oxygen. Sometimes this list includes magnesium and sulfur. Without considering the special role of such isotopes as D, $^6$Li and $^{10}$B in the defense industry and nuclear power engineering, attention should be paid to the use of stable isotopes of light elements, mainly in the medical and biological fields.

At the same time, for some isotopes that have become instruments of large-scale clinical diagnostics, it is necessary to consider the change in the volume of production for a long time, and in particular for the last 10-20 years, as well as the technologies which are used for isotope enrichment.
2.2. Main fields of uses
In general, it is possible to single out, as noted in [1], six main directions for the use of stable isotopes. The first is earth sciences, that is, geochemistry, geology, geophysics, hydrology, and marine sciences. The second: biochemistry, biology, biotechnology, chemistry, physics, physiology, that is, fundamental and applied research. The third is food and agriculture. The fourth is nutrition. The fifth is toxicology. And, finally, health care. However, it is not difficult to see that the last five regions are combined and in one way or another connected with the last direction - health care.

2.3. Healthcare application
The application of stable isotopes of light elements in health care should be divided into a number of special areas. First of all, it is medicine, biology and pharmacology. The most common specific applications are listed in Table 1 [1-3], where is reflected the special role of $^{13}\text{C}$ (the breath tests for diagnosis of $H.\ Pilory$ infection) and $^{18}\text{O}$ (the starting material in the production of $^{18}\text{F}$ for positron emission tomography) as diagnostic isotopes in clinical medicine [3-6].

Table 1. The main directions of the application of stable isotopes of light elements in the field of medicine, pharmacology and biology.

| Isotope      | Application                                                                 |
|--------------|------------------------------------------------------------------------------|
| Deuterium    | Vitamin research                                                             |
|              | Noninvasive, accurate, and prolonged measurement of energy expenditures during |
|              | everyday human activity                                                      |
| Lithium-7    | Starting material for berylliosis studies ($^7\text{Be}$)                     |
| Boron-10     | Boron neutron capture therapy (BNCT)                                         |
| Boron-11     | Starting material for positron emission tomography (PET diagnosis, $^{11}\text{C}$) |
| Carbon-13    | Noninvasive breath tests for metabolic research and diagnosis ($H.\ Pilory$ infection) |
|              | NMR research and diagnosis                                                   |
| Nitrogen-15  | Excrement test for $H.\ Pilory$ infection                                   |
| Oxygen-17    | Measurement of the blood stream velocity                                    |
| Oxygen-18    | Noninvasive, accurate, and prolonged measurement of energy expenditures during |
|              | everyday human activity                                                      |
|              | Starting material for positron emission tomography (PET diagnosis, $^{18}\text{F}$) |

a Research, not clinical practice

In addition to the applications indicated in the table, both proteomics and clinical pharmacology should be indicated, such as the assessment of drug products or drug delivery systems, assessment of patients and assessment of drug pharmacology, as well as numerous studies aimed at the prospect of use in clinical practice, including new opportunities of $^{13}\text{C}$-breath tests [6-9].

3. Scale of production and its changes

The technology of stable isotopes of light elements and their production have a relatively short time interval - less than 100 years. The very discovery of many stable isotopes dates from 1913-1920 [10]. Naturally, experimental-industrial installations for the enrichment of isotopes began to be created in the 1930s-1940s [11]), and the first grams of highly enriched $^{13}\text{C}$ (99.8% at.), $^{15}\text{N}$ (99.8% at.) and $^{18}\text{O}$ (99.75% at.) were obtained in the 50s of the XX century in K. Clusius laboratory by the thermal diffusion method [12]. Therefore, considering the change in the production scale of stable isotopes in
time, the starting point can be taken in 1940, when the output was estimated at hundreds of grams [11]. Twenty years later, by 1960, isotope production could provide tens of kilograms, and approximately in 1980 the total scale of production was estimated at about 100 kg annually and by the beginning of the third millennium overcome the 1,000 kilogram mark (Fig. 1). In this case, the dependence of the production volume of stable isotopes of light elements, more precisely $^{13}$C, $^{15}$N and $^{18}$O, in semilogarithmic coordinates can be represented by a straight line with a high correlation coefficient $\approx 0.98$.

**Figure 1.** The production scale of $^{13}$C, $^{15}$N and $^{18}$O highly enriched stable isotopes

As follows from the figure, every 20 years there is an increase in the global production volume by approximately one order of magnitude, that is, 10 times. It can be calculated that annually the production volume ($\Delta V_{an}$) increases on average by

$$\Delta V_{an} = \frac{t-1}{2} \times 100 = \frac{10^{-1}}{2} \times 100 \approx 2 \%$$

and this increase is characterized by an enviable constancy. The forecast for the change in the volume of the global market for stable isotopes of light elements and labeled compounds for the next 5 years to 2022 points to 2.94% of the annual increase and the reaching of $294.2$ million at the end of this period as noted on several websites. It is noted that the main factors that stimulate the growth of this market are an increase in research activities in the pharmaceutical and biotechnological sectors, the growth of research in proteomics and the expansion of the incidence of cancer. However, as noted repeatedly, the high cost of labeled compounds will to some extent impede market growth during the forecast period. Nevertheless, the history of production development and the forecast for the next five years show an exponential growth in the production and consumption of isotope production, which is clearly shown in Fig. 2 for isotopes of $^{13}$C and $^{18}$O.

It should be noted that for the last 10-15 years a new quantitative and qualitative transition has occurred – the volume of production has overcome the tonnage boundary (Fig. 1, 2a). It can assume that the $^{13}$C and $^{18}$O isotopes from the group of "research isotopes" passed into a new group of "diagnostic". It is interesting to note the decline in production of $^{18}$O in the period 1980-90, which is clearly seen in Fig. 2b. The most likely explanation is the stop of ICON production in the Los Alamos laboratory in the US, as well as the accident of the installation of low-temperature rectification of NO in the company Isotec, Inc., which led to a deficit in the market for heavy-oxygen water in this period of time.
Figure 2. Change in the annual production volume of $^{13}\text{C}$ (●) and $^{18}\text{O}$ (○) isotopes by years: $a$- on an ordinary scale; $b$- in semilogarithmic coordinates

4. Manufacturers and technologies

For the two "diagnostic" isotopes $^{13}\text{C}$ and $^{18}\text{O}$ (in the form of heavy-oxygen water), their manufacturers and the technologies used are shown in Tables 2 and 3 respectively, and Figures 3 and 4 reflect the contribution of this or that technology to the total production volume of isotope products. Analysis of the tables and figures shows that the dominant amount of light isotopes is produced by physicochemical methods.

Producers and technologies for the $^{15}\text{N}$ isotope are considered separately in Table 4. Production of this isotope for many years remains at the level of several tens of kilograms annually and the main production volumes based on the chemical exchange between nitrogen oxides and nitric acid (Nitrox process).
### Table 2. Production of $^{13}$C in the world.

| Country | Manufacturer | Method of production | Volume, $^{13}$C kg C/an |
|---------|--------------|----------------------|--------------------------|
| USA     | Isotec, Inc. | CO Low-temperature rectification | 100 |
|         | CIL, Inc.    | CO Low-temperature rectification | 420 |
| China   | Jiangsu Zhengneng Isotope Co., Ltd. | CO Low-temperature rectification | Project 600 |
|         | RRC “KI”$^a$ | CO Low-temperature rectification | Stand by |
| Russia  | Gas-Oil, Ltd. SC “PA Electrochemical Plant” | Laser / Gas centrifugal separation | Does not exist |
|         | ICON, LANL   | Gas centrifugal separation | ≈ 5 |
| Japan   | Tokyo Gas Company | CH$_4$ (LNG) Low-temperature rectification | 100 |

$^a$ Russian Research Center "Kurchatov Institute"

### Table 3. Production of heavy-oxygen water H$_2^{18}$O in the world.

| Country          | Manufacturer | Method of production | Volume, H$_2^{18}$O kg O/an |
|------------------|--------------|----------------------|----------------------------|
| Romania          | INCDTIM$^a$  | Chemical Exchange NO-HNO$_3$ | ? |
| Georgia          | NHTCG$^b$    | NO Low-temperature rectification | 10 |
| USA              | Isotec, Inc. | NO Low-temperature rectification | 120 |
|                  | CIL, Inc.    | Vacuum rectification of water | 430 |
|                  | ICON, LANL   | NO Low-temperature rectification | Stand by |
|                  | Marshall Isotopes, Ltd. | Vacuum rectification of water | 150 |
|                  | Rotem Industries Ltd. | Vacuum rectification of water | 220 or 250 |
| China            | SRICI$^c$ Co., Ltd. | Vacuum rectification of water | 100 |
|                  | Jiangsu Zhengneng Isotope Co., Ltd. | Vacuum rectification of water | 400 |
| Russia           | CJSC “GST”$^d$ | Vacuum rectification of water | 60-80 |
| South African Republic | Klydon | Vacuum rectification of water | 200-230 |
| Japan            | Taiyo Nippon Sanso Corp. | O$_2$ Low-temperature rectification | 600 |

$^a$ National Institute for Research and Development of Isotopic and Molecular Technologies
$^b$ National high technology centre of Georgia;
$^c$ Shanghai Research Institute of Chemical Industry;
$^d$ CJSC “Global Scientific Technologies”
Figure 3. $^{13}$C isotope technologies and their share in the total production scale

Figure 4. The isotope $^{18}$O technologies and their share in the total production scale (in the form of $H_2^{18}$O)

Table 4. Production of $^{15}$N in the world.

| Country   | Manufacturer                      | Method of production                        | Volume, kg $^{15}$N/year |
|-----------|-----------------------------------|--------------------------------------------|---------------------------|
| Georgia   | NHTCG $^a$                        | Chemical Exchange NO-HNO$_3$                 |                           |
| USA       | Isotec, Inc.                      | NO Low-temperature rectification            |                           |
| USA       | CIL, Inc.                         | NO Low-temperature rectification            | 16                        |
| Romania   | INCDTIM $^b$                      | Chemical Exchange NO-HNO$_3$                | Stand by                  |
| China     | SRICI $^c$, Ltd.                  | Chemical Exchange NO-HNO$_3$                | 30                        |
| Japan     | SHOKO Science Co., Ltd. (SI Science Co., Ltd.) | Chemical Exchange NO-HNO$_3$ | 5                         |

$^a$ National High Technology Centre of Georgia;
$^b$ National Institute for Research and Development of Isotopic and Molecular Technologies
$^c$ Shanghai Research Institute of Chemical Industry

Attention is drawn to the production of $^{13}$C in Russia (Table 3) where, in contrast to other countries of the world, physical separation methods are developing - laser and gas centrifugal. In this case, the first one does not allow economical production of highly enriched $^{13}$C $^{[13]}$, and the second one is characterized by a relatively low productivity $^{[14]}$. In addition, the combination of these two methods, when used consecutively for the production of highly enriched heavy carbon isotope $^{[15]}$, is likely to lead to expensive $^{13}$C, which can not contribute to the widespread introduction of diagnostic procedures with its use in the practice of clinical medicine. The separation of light-mass isotopes is most economical when using physicochemical separation methods (rectification, chemical isotope exchange). Existing publications $^{[16, 17]}$ demonstrate this clearly, and gas centrifuging, for example, is theoretically characterized by two orders of magnitude of higher energy costs compared to the process of rectification $^{[18]}$. 
5. Features of technological development of isotope separation of light elements

5.1. Analysis of technological changes

Analysis of the technologies used for separating carbon isotopes on a large scale shows that, along with the relatively recently used CO low-temperature rectification, the enrichment coefficient $\varepsilon \approx 7 \times 10^{-3}$, a new methane rectification technology appeared. The $^{13}$C methane distillation plant has been in operation since 2000 as a result of lengthy studies initiated in the first half of the 1990s by the Japanese company Tokyo Gas, which produces liquefied natural gas (LNG) [19]. But the main thing is an example of the production realization of the physicochemical isotope separation process, characterized by the value of the enrichment factor $\varepsilon \approx (3-4) \times 10^{-3}$. Other similar technologies were not implemented either for rectification or for chemical exchange to the company Tokyo Gas.

In the case of $^{18}$O enrichment, the dominant technology is rectification of water under vacuum. At the same time, the transition from an environmentally dangerous technology is seen (NO low-temperature rectification) to a more pure process, which is the rectification of molecular oxygen.

5.2. New opportunities

Technological successes achieved in Japan allow us to optimistically consider the possibility of producing a heavier nitrogen isotope $^{15}$N, which can be claimed as a component of nitride fuel in nuclear power engineering in fast neutron reactors [20]. In addition, an increase in the volume of its production will undoubtedly lead to a spreading of the application of $^{15}$N in medicine and biology (Table 1). Features of large-scale production of $^{15}$N were considered repeatedly [16, 21], but the technologies used are the chemical exchange between NO, NO$_2$ and HNO$_3$; NO low-temperature rectification and the methods developed [17, 22, 23] are either environmentally hazardous or economically unsuitable, which is probably explained by the low production of $^{15}$N. The most environmentally friendly and therefore scalable technology can be a low-temperature rectification of molecular nitrogen, realized by a transit scheme in combination with air separation plants producing liquid nitrogen. The value of the enrichment factor in such a process is at the same level as in the rectification of methane, $\varepsilon = 4 \times 10^{-3}$ at $T = 77$ K [11]. To obtain a highly enriched $^{15}$N, such a process must provide for a homomolecular isotopic exchange (HMIEX)

$$2^{14}\text{N}^{15}\text{N} \rightleftharpoons ^{14}\text{N}^{14}\text{N} + ^{15}\text{N}^{15}\text{N}$$

and the stage of additional enrichment of $^{15}$N at the extraction of lighter molecules of $^{14}$N$^{14}$N from nitrogen, as is the case with the production of $^{13}$C by CO low-temperature rectification [11, 24]. One of the main problems in the use of physicochemical processes with a small value of $\varepsilon$ – is a large steady-state time, possibly measured in years.

In order to overcome this disadvantage, it is proposed, at the initial stage of $^{15}$N enrichment, to fill the distillation columns by the previously $^{15}$N enriched and produced using the isotope separation method, which is most effective from the single isotopic effect point of view, that is, using the Nitrox process. For this purpose a starting small-scale installation is created, producing $^{15}$N with a concentration (3-30)\% at. in the form of a mixture of NO and NO$_2$, which is then easily reduced by hydrogen to molecular nitrogen on the catalyst

$$\text{NO, NO}_2 + \text{H}_2 \rightarrow \text{N}_2 + \text{H}_2\text{O}$$

The formed nitrogen passes through a stage of deep drying, is liquefied and poured into the columns, forming in them a fluid hold-up enriched by $^{15}$N (Fig. 5). Calculations show that these techniques should ensure that the steady-state time decreases by approximately an order of magnitude. The proposed scheme can be considered as a separation process, based on the joint use of two physicochemical methods for isotope separation.

The development of physicochemical methods is based not only on their combination, but also on the application of new working systems in the chemical isotopic exchange, and on the use of a new type of equipment. Let us consider these two directions, for example, at the separation of boron isotopes. It is known that the production of boron isotopes is based on chemical isotope exchange between gaseous BF$_3$ and its liquid molecular complex, for example, with CH$_3$OC$_6$H$_5$ (methylphenyl
ether or MPE) in mass-exchanging columns with a height of ≈ 60 m [25].

**Figure 5.** The large-scale production of $^{15}$N with starting unit (production of low enrichment $^{15}$N by Nitrox process) for the formation of $^{15}$N hold-up in columns for low-temperature rectification of N$_2$: SU – starting unit; CB – conversion block (восстановление смеси NO и NO$_2$ до N$_2$ и глубокая осушка N$_2$); SM 1 … SM N – separation modules for $^{15}$N enrichment by N$_2$ rectification.

This requires the use of high production facilities, which leads to large capital and operating costs. The solution of the problem is the use of horizontal rotor mass-exchange columns, which have the same efficiency in comparison with classical vertical equipment. In Fig. 6 shows the results of a horizontal rotor column tests both in rectification mode and in the regime of chemical isotope exchange at the separation of boron isotopes [26]. In the latter case, based on the achieved $S$ value, the number of theoretical separation plates (NTP) turned out to be equal 22±2, and the height of the theoretical plate was (3.6±0.4) cm.

**Figure 6.** The results of a horizontal rotary column tests with 80 cm long and spiral-prismatic packing 2.5×2.5×0.2 mm: a- change the separation degree of the components of the model mixture in the rectification mode without rotation and with rotation of the mass transfer part; b- kinetic curve for separation of boron isotopes in the regime of chemical exchange between BF$_3$ and its complex with MFE

More impressive results were obtained with the first experimental separation of boron isotopes between BF$_3$ and its complex with nitromethane CH$_3$NO$_2$ [27-29], that is, in the two phase system, the singly stage separation factor in which is measured in [29]. In a column with a height equal to only 1.4 m, the value of the separation degree was achieved $S = 3.8$ (Fig. 7) with a difference in $^{10}$B concentration at the ends of the column equal to ≈ 30% at. [28]! It is interesting to note that the same separation degree was achieved by rectification of BCl$_3$ in an experimental plant with columns of a total height of 22 m [30].
Figure 7. The change of the separation degree of boron isotopes during the chemical exchange between BF₃ and its complex with CH₃NO₂ in a mass-transfer column with 140 cm height and a spiral-prismatic packing of 1.2×1.2×0.2 mm

This more than clearly demonstrates the advantage of the chemical exchange method at the separation of boron isotopes in comparison with the rectification method and, moreover, in comparison with the physical methods of isotope separation.

6. Conclusion

During the last 15-20 years there have been significant changes in the production of stable isotopes of light elements. The production scale of ¹³C and ¹⁸O isotopes is measured in thousands of kilograms that was achieved by the development and use of physicochemical methods of isotope separation. These methods - rectification and chemical isotope exchange - have been developing in the world and Russia. Both new working systems and a new type of equipment were studied, new technical solutions were proposed to create more cost-effective technologies for production of stable isotopes of light elements.

7. References

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