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UTILIZATION OF NUCLEAR METHODS FOR MATERIALS ANALYSIS AND
THE DETERMINATION OF CONCENTRATION GRADIENTS

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16. Abstract The various types of nuclear chemical analysis methods are discussed. The possibilities of analysis through activation and direct observation of nuclear reactions are then described. Such methods make it possible to analyze trace elements and impurities with selectivity, accuracy, and a high degree of sensitivity. Such methods are also useful in measuring major elements present in materials which are available for analysis only in small quantities. Moreover, these methods are well suited to superficial analyses and to determination of concentration gradients, provided the nature and energy of the incident particles are chosen judiciously. The report is illustrated with typical examples of steels, pure iron and refractory metals.			
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UTILIZATION OF NUCLEAR METHODS FOR MATERIALS ANALYSIS AND THE DETERMINATION OF CONCENTRATION GRADIENTS

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The various types of nuclear chemical analysis methods are discussed. The possibilities of analysis through activation and direct observation of nuclear reactions are then described. /345*

Such methods make it possible to analyze trace elements and impurities, even those present in very slight quantities, with selectivity, accuracy and a high degree of sensitivity. The latter quality also makes such methods useful in measuring major elements present in materials which are available for analysis only in small quantities. Moreover, these methods are well suited to superficial analyses and to determination of concentration gradients, provided the nature and energy of the incident particles are chosen judiciously.

The report is illustrated with typical examples of analyses of steels, pure iron and refractory metals.

Introduction

Generally speaking, chemical analysis consists of identifying and measuring an element or compound by isolating it and noting its reaction to an appropriately selected external stimulus, among which the most traditional is a chemical reagent.

Nuclear methods use various nuclear radiations as "reagents". These radiations consist of neutrons, charged particles (protons, deuterons, helions) or gamma photons, whose effects on a given substance are analyzed. Nuclear analysis methods can be placed in three categories:

*Numbers in the margin indicate pagination in the foreign text.

Methods without Nuclear Reactions

- Detection of a modification in direction, intensity or type undergone by a given incident radiation, such as partial absorption, elastic or non-elastic diffusion, X-ray emission or neutron diffraction.

Methods with Nuclear Reactions

- Analysis through activation: detection (after irradiation) of typical radiation emitted by a radioactive element generated upon irradiation of the substance, issuing from the element to be measured.
- Direct observation of nuclear reactions: detection of secondary radiation characteristic of the element to be measured, as determined during irradiation of the substance by means of appropriate incident radiation.

Methods which require Use of Radioactive Tracers

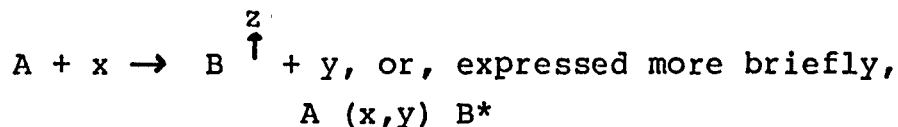
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- Analysis by means of isotopic dilution with a known quantity of a pure radioactive constituent.
- Analysis by means of a radioactive chemical reagent.
- Radiometric titration.

The methods mentioned in the first category above are predominantly qualitative or semi-qualitative. The methods listed in the third category apply only to a small number of particular cases. Taking into consideration the space and time available, we will limit our discussion to methods of analysis by activation and direct observation of nuclear reactions, since these are the most generally and frequently applicable methods.

A distinction should be drawn between the two latter methods, since these fundamentally different methods are sometimes bracketed under the same rubric of "analysis by activation." A nuclear reaction in fact takes place:¹

¹Example: $^{59}\text{Co} (n, \gamma) ^{60}\text{Co}$ emitting γ'
 $^{11}\text{B} (p, n) ^{11}\text{C}$ emitting γ''



where

A = a stable element

B* = a radioactive element (emitting radiation z)

x = incident radiation

y = prompt radiation characteristic of the nuclear reaction

z = secondary radiation characteristic of the radioactive element formed, which is itself determined by the initial element and the radiation received by the initial element.

The y and z radiations are characteristics of the element and the incident radiation. The z radiation plays a part in analysis through activation properly so-called, while the y-radiation relates to direct observation of nuclear reactions.

It should also be noted that since only atomic nuclei are involved in nuclear reactions, the analytical methods must be exclusively elementary. On the other hand, it is important to recall that nuclear reactions generally occur only at given energy levels, i.e., at their energy thresholds.

Analysis by Activation

1. Except in special cases, the sample is given no special treatment prior to irradiation.

The sample to be analyzed is irradiated for a suitable length of time by a stream of neutrons, charged particles or gamma photons at an energy level sufficient to cause formation of radioactive elements by means of one or several nuclear reactions within the element to be measured.

After irradiation, the activity level of the sample is compared to that of an irradiated control. This simultaneous comparison allows the concentration of the subject element to be measured. The activity is measured by means of beta or gamma detectors or with high-performance spectrometers.

The characteristics of the radiation emitted by a radioactive element are particular to the atom under consideration. The type, energy and decay of this radiation as a function of time related to its period, allow the emitting radioactive element to be characterized (qualitative analysis). Furthermore, measurement of the radiation flow produced allows the element itself to be measured (quantitative analysis). In practice, the spectra obtained are analyzed. This procedure is becoming more and more automatic through connection of an on-line computer to the spectrometer channel. Briefly, the peak energy identifies an element, and the level of the peak indicates the quantity of the element present (Figure 1).

Depending on the choice of incident particles on the one hand, and on their energy with respect to the energy threshold of the nuclear reaction on the other hand, it is often possible to avoid matrix interference. In this manner the analysis is purely instrumental and depends exclusively on spectrometry of gamma or beta radiation.

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Under other circumstances, i.e., when other elements present in the sample are too active with respect to those elements which are to be measured, chemical separations are carried out after irradiation (Figure 2). A major advantage of the activation analysis method is due to the fact that after irradiation, samples can be handled without taking any special precautions, since contamination which may occur at this stage will not affect the analysis, which depends only on the radioactive elements produced during the preceding irradiation (Figure 3).

If there is particular interest in the surface of the samples, then lightly rinsing the samples after irradiation will tend to prevent errors which may occur due to contamination prior to or during irradiation.

The above-mentioned advantage is especially important in measuring the presence of oxygen, nitrogen or carbon in metals. Thus, combustion methods, or reductive or carburetting fusion (which are generally used in making such measurements) can be used here in a rudimentary manner. Contamination of samples by air or by carbonous reagents has no effect on analytic results, since the analysis is concerned only with a specific radioactive isotope produced during irradiation, such as for example carbon-11, fluorine-18, oxygen-15 or nitrogen-13. This advantage is obviously even more useful when the reactivity of of the metal increases (Table 1).

Inactive entraining agents which do not affect the analytic results may also be added. Such agents tend to minimize errors due to possible losses during chemical operations.

2. In general, neutronic activation is used primarily with relatively heavy elements, while activation by means of charged particles or gamma photons is reserved for light elements (up to fluorine or sulfur). But the latter method is also useful in measuring some of the heavy elements such as lead, thorium, bismuth, titanium, iron, nickel and zinc.

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The detection sensitivity of the neutronic activation method (Table 2) and the photonic activation method (Table 3) are currently on the level of parts per million. The detection sensitivity of the charged-particle activation method approaches parts per billion (Table 4). Under favorable circumstances, these limits are much lower. As a result, samples with small mass (on the order of several milligrams) may suffice. In other words, the activation analysis method is extremely sensitive.

3. Finally, analysis by activation of charged particles is particularly well suited to superficial determinations, due to the progressive slowing of these particles upon penetration of the subject material (Figure 4). It is necessary only to select the energy of such particles so that nuclear interreactions do not occur beyond a given, narrow thickness, which allows the thickness of the sample in question to be determined. While the detection limits are usually on the order of 10^{-2} micrograms/cm², these limits can reach 10^{-5} to 10^{-6} micrograms/cm² under favorable circumstances.

Analysis by Direct Observation of Nuclear Reactions

This method involves the prompt radiation issuing directly from the nuclear reaction, not only that emitted a posteriori by a radioactive element.— The incident particles utilized are essentially charged particles (especially protons or deuterons) or gamma photons, and the radiation detected may comprise photons, protons or alpha particles, or even fluorescent X-rays. Therefore, the analysis is in principle instantaneous, as opposed to analysis by activation strictly so-called.

The analytic apparatus is aligned with the path of the beam of the accelerator utilized, which in most instances is a Van de Graaff providing low-energy particles of from 2 to 5 MeV. The operation should be carried out under reduced pressure (Figure 5).

Nevertheless, it is important to note that some nuclear reactions are distinguished by the presence of resonances without which their effective sections are substantially nil. These resonances are narrow, and such that when the energy of incident particles is increased beyond the resonance value (taking into account the slowing of the charged particles upon penetration of the substance), the resonance level is reached at the end of a path whose depth increases as the initial energy is increased. Thanks to this phenomenon, it is possible to carry

out superficial analyses and also to determine concentration profiles near the surfaces of solids by entirely non-destructive means. This method is effective for thicknesses of material on the order of 1 to several microns.

The performance of the techniques described above is characterized by detection limits which are currently on the order of about 10^{-3} micrograms/cm² of superficial carbon or nitrogen (Table 5). Accessible depths attain 10 micrometers, with respective depth resolutions of 0.5 and 0.01 micrometers.

Mention should also be made of the X-fluorescence induced by the action of low- or medium-energy charged particles (from a few keV to several MeV). This method is extremely sensitive with respect to multi-element analysis of narrow layers (layers several microns thick) comprising medium or heavy elements.

Typical Applications

Analysis of Steels

For example, in austenitic steels, the majority of the medium-weight or heavy elements can be measured through neutronic activation. Cobalt (at 30 to 1500 ppm) and manganese (from 100 ppm to 1%) are currently measured by entirely instrumental means in samples on the order of a gram (Figure 6). Arsenic (at 10 to 600 ppm) is determined from its sulfide S_1As_2 , and phosphorus (at 50 to 100 ppm) is determined after precipitation of ammonium magnesium phosphate.

Among the light elements, oxygen can be measured through fast-neutron activation (at an energy level of 14 MeV) by means of neutrons emitted by a portable neutron generator.²

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²Nuclear reactors provide essentially thermal neutrons, with high flow rates (up to 10^{15} n/cm² s⁻¹), which entail extremely pronounced analytic sensitivity. With respect to radiation sources, the latter have the advantage of being portable, but produce only limited neutron flows ($\sim 10^0$ n/cm² s⁻¹). It should also be noted that fast neutrons cause much more interference between the various elements comprising the sample.

With respect to these 14-MeV neutron generators, it should be noted that an on-line analysis system is currently in operation in a Belgian steel plant. This system produces 400 oxygen analyses of steel per day, up to 1 mg in a 40-g sample. (The $^{16}\text{O}(n,p)^{16}\text{N}$ reaction has a period of 7.4 seconds.) Other on-line analysis operations are being developed with isotopic sources of californium-252.

The light elements are treated more specifically and with greater sensitivity through charged-particle or gamma photon activation (Table 6).

Direct observation of nuclear reactions allows measurement of carbon and nitrogen concentration gradients (Figures 7 and 8). Under the influence of protons and by detection of prompt gamma rays, the thicknesses which can be analyzed non-destructively can be as great as 10 micrometers, with 0.5-micrometer resolution for carbon and 0.01-micrometer resolution for nitrogen. Under the influence of deuterons and through detection of protons or alpha particles, the resolution is on the order of a micron. These two variants are thus complementary.

Gradients of fluorine (Figure 9) or chlorine are determined in an analogous manner, depending on the depth. It should be noted, however, that the depths analyzed may be accrued by means of successive abrasions (Figure 8).

Superficial analysis also makes it possible to measure the variations of concentrations along the length of a tube (in problems connected with cleaning metals, for example), by turning the tube in front of the incident beam (Figure 10).

Analysis of Corrosion Products

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The water or steam conduits in light-water nuclear reactors are fabricated of Inconel and 18 Cr/10 Ni austenitic steel. While corrosion of these alloys is slight under the circumstances in question, particles may still be released into the cir-

culating water. It is worthwhile to ascertain the composition of these particles, but only slight quantities (several mg) can be recovered. The analysis of these particles has been carried out through neutronic activation (Table 7). It will be seen that major elements and impurities can easily be measured at the same time.

Analysis of Pure Metals

Activation analysis is the most reliable method of measuring the slightest traces of impurities in high-purity metals, using simple chemical separations. For example, the last remaining impurities in a "pure" iron were measured according to this method (Table 8).

Oxygen, carbon and nitrogen can also be measured in an extremely sensitive and reliable manner, through activation of gamma photons or of helions-3 for nickel, cobalt or silicon (Table 9).

Analysis of Refractory Metals

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With traditional methods it is especially difficult to measure nitrogen in metals such as molybdenum, and even more so in metals such as tantalum or tungsten, whose nitrogen content is slight but nevertheless significant with respect to the properties of these metals. The problem can be solved through activation with gamma photons or helions-3 (Table 10).

Generally speaking, other methods yield substantially less accurate results, especially in the case of oxygen, due to the effect of residual impurities.

Conclusions

It is worthwhile to point out the analytic advantages and drawbacks of nuclear techniques. Analysis by activation (AA) and analysis by direct observation of nuclear reactions (ANR) are often criticized because of their relatively mediocre pre-

cision, which is currently from 5 to 10%. Nevertheless, when special care is given to operating conditions (extended irradiation and/or calculation periods), it is possible to obtain accuracy on the order of 1 to 3%. This degree of accuracy is especially attainable for repetitive analyses or analyses of long series of samples. The above-mentioned degrees of accuracy remain valid for trace analyses. It is currently easily possible to measure from 1 part per billion to nearly 10%.

In summary, numerous advantages of nuclear methods are generally acknowledged:

- High sensitivity and extremely low detection limits
- Insusceptibility to contamination during chemical treatments after irradiation (i.e., superficial contamination)
- Absence of residual traces in equipment and/or impurities in chemical reagents
- Direct and primary sampling (regardless of the chemical state of the element to be measured)
- Reference methods
- Possibility of non-destructive analysis
- Ease of automatization.

In fact, thanks to their various forms, nuclear methods make it possible to solve most of the problems of elementary analysis. Their preferred areas of application, with respect to their individual performances, are the following:

- Trace analysis
- Multi-elementary analysis
- Measurement of certain elements in complex matrices, or elements comprising multiple compounds
- Analysis of major elements in very small quantities of material (e.g., corrosion products and/or dusts)
- Superficial analyses
- Measurement of concentration gradients in limited thicknesses of material.

Acknowledgements

The analytic methods described in this report were researched, put into operation and studied at the Saclay Center for Nuclear Research by two teams in the Nuclear and Isotopic Chemistry Research and Analysis Division (SAECNI): the Neutronic Activation Analysis Group (using facilities of the Pierre Sue Laboratory) and the Nuclear Reaction Analysis Laboratory, headed respectively by S. May and C. Englemann.

The author wishes to express his thanks to the laboratory directors as well as to their colleagues for their contributions to this report, especially with respect to the results of typical analyses which they provided for purposes of illustration.

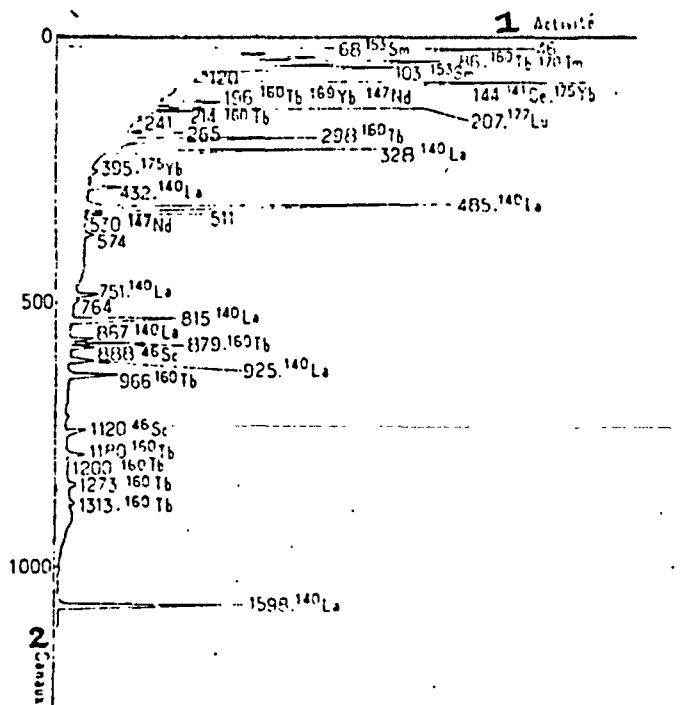


Figure 1. Rare earth spectrum in graphite after neutronic irradiation, decay period of 8 days and detection in a Ge(Li) sample. Peak energy in keV.

Key:

1. Activity
2. Channels

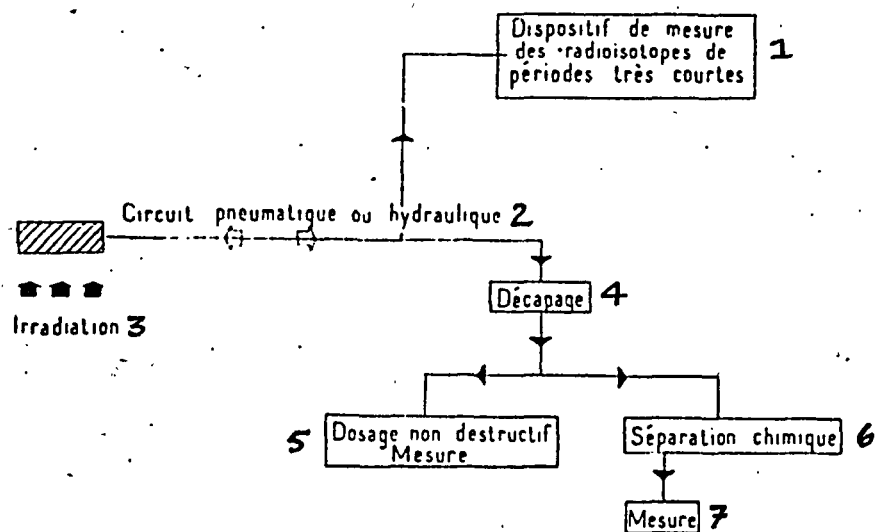


Figure 2. Principal stages in activation analysis

Key:

1. Measuring apparatus for isotopes with very short half-lives
2. Pneumatic or hydraulic circuit
3. Irradiation
4. Cleaning
5. Non-destructive sampling; measurement
6. Chemical separation
7. Measurement

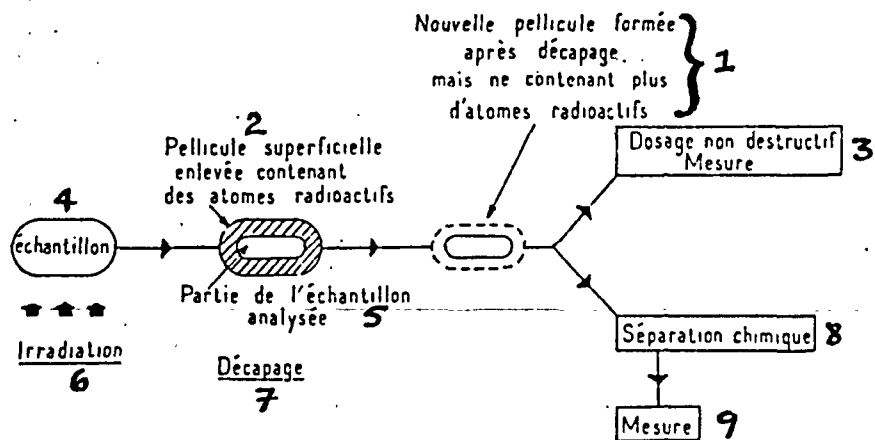


Figure 3. Role played by cleaning of samples after irradiation

Key:

1. New film formed after cleaning, containing no radioactive atoms
2. Removal of superficial film containing radioactive atoms
3. Non-destructive sampling; measurement
4. Sample
5. Portion of sample analyzed
6. Irradiation
7. Cleaning
8. Chemical separation
9. Measurement

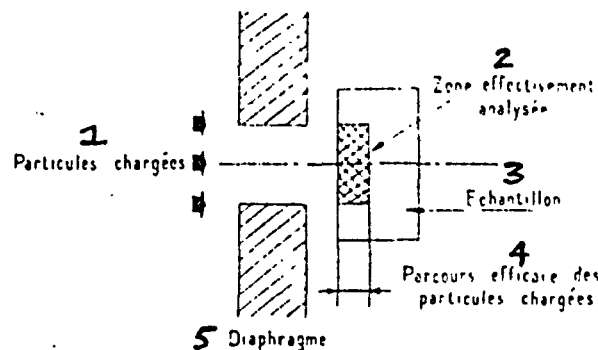


Figure 4. Activated zone of a thick sample irradiated by charged particles

Key:

- | | |
|------------------------------|--|
| 1. Charged particles | 4. Effective path of charged particles |
| 2. Zone effectively analyzed | 5. Diaphragm |
| 3. Sample | |

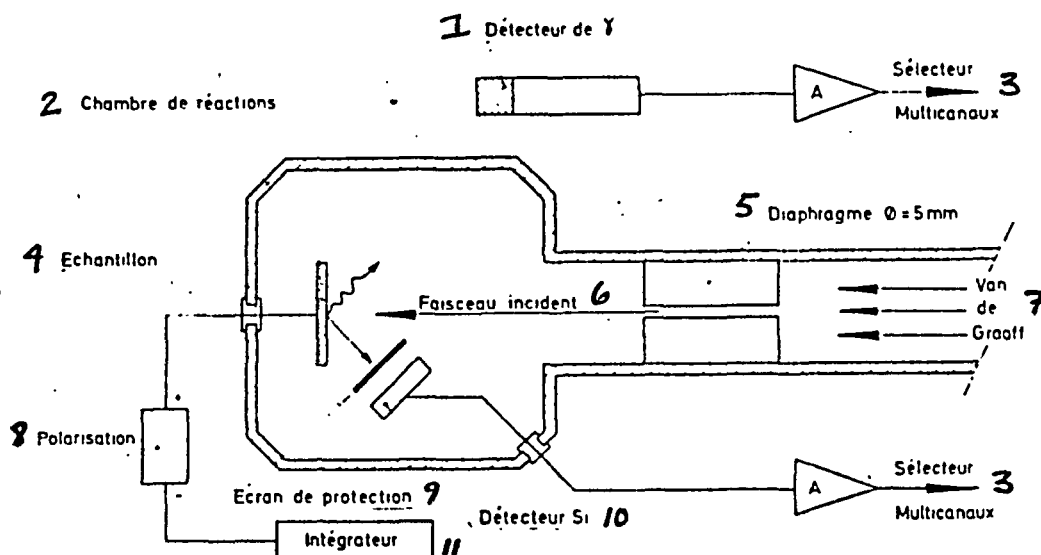


Figure 5. Diagram of apparatus for superficial analysis by direct observation of nuclear reactions

Key:

- | | |
|--|----------------------|
| 1. Gamma-ray detector | 7. Van de Graaff |
| 2. Reaction chamber | 8. Polarization |
| 3. Multichannel selector | 9. Protective shield |
| 4. Sample | 10. Si detector |
| 5. Diaphragm ($\varnothing = 5\text{ mm}$) | 11. Integrator |
| 6. Incident beam | |

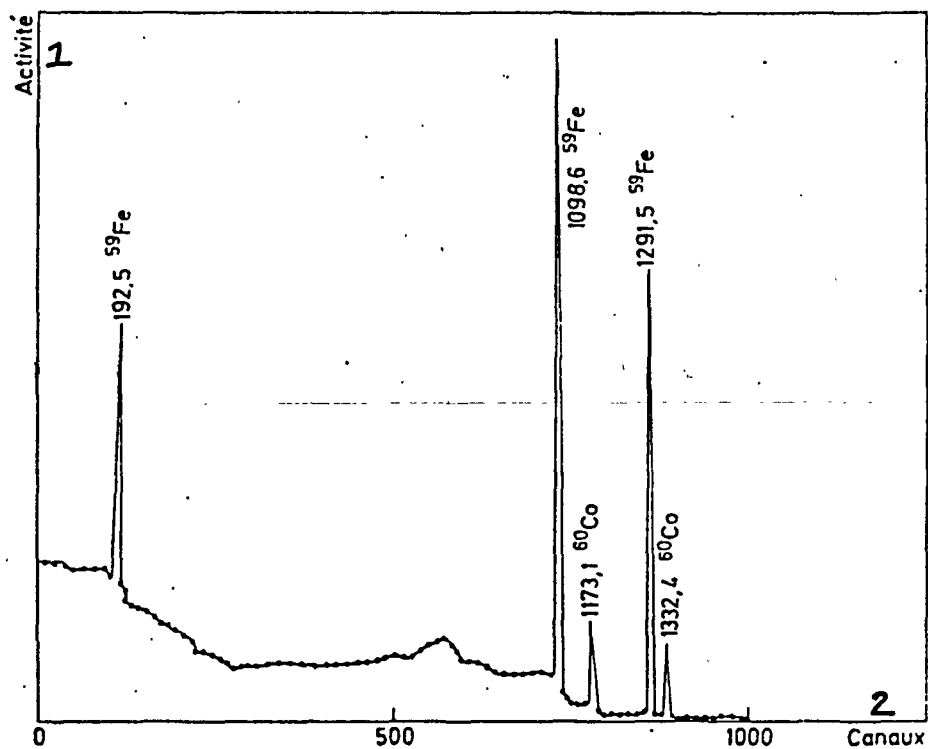


Figure 6. Analysis of cobalt in steels by neutronic activation and detection in a Ge(Li) sample. Peak energies are indicated in keV.

Key:

1. Activity
2. Channels

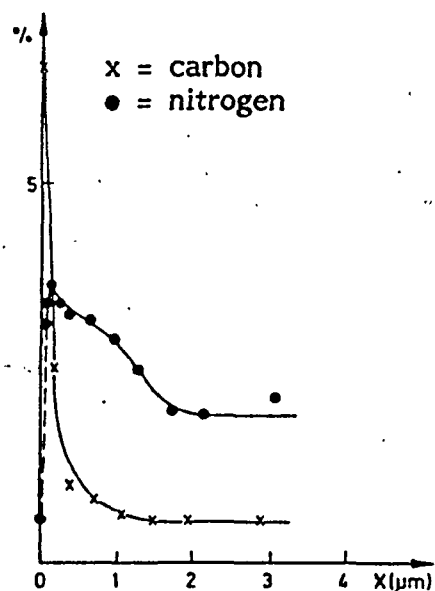


Figure 7. Examples of concentration profiles of carbon and nitrogen below the surface of a steel sample, determined through resonant nuclear reactions: $^{12}\text{C}(p, \gamma)^{13}\text{N}$ and $^{15}\text{N}(p, \alpha\gamma)^{12}\text{C}$

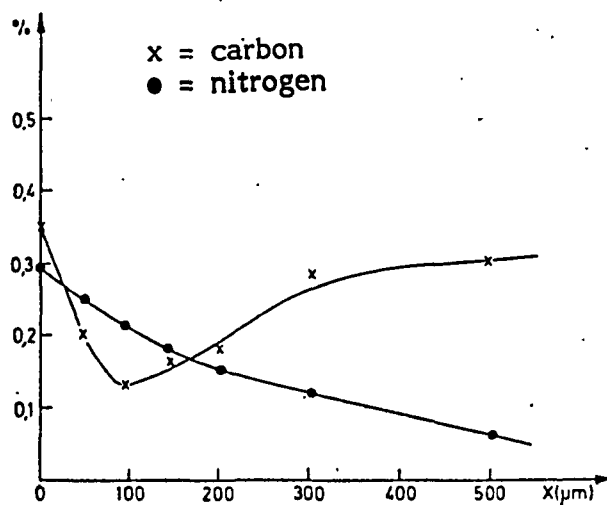


Figure 8. Examples of carbon and nitrogen concentration profiles according to the thickness of a sample of carbonitrated steel, determined through nuclear reactions: $^{12}\text{C}(d, p)^{13}\text{C}$ and $^{14}\text{N}(d, \alpha)^{12}\text{C}$. In this case, successive layers were removed by machining on a precision lathe.

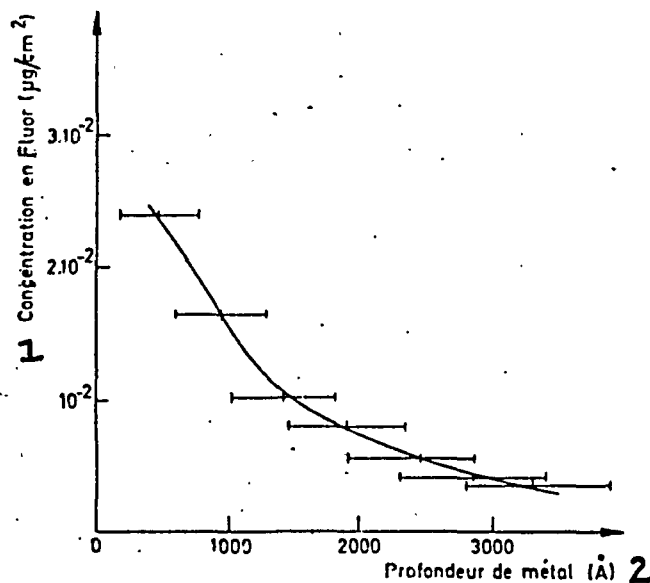


Figure 9. Determination of fluorine concentration gradient from the surface of iron, by detection of prompt gamma photons emitted during proton irradiation

Key:

1. Fluorine concentration

2. Metal depth

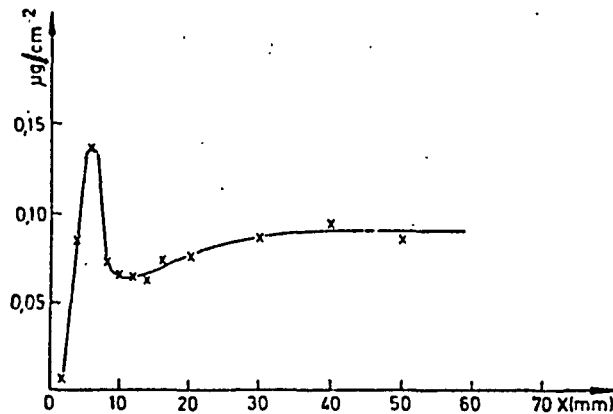


Figure 10. Example of curve representing variation of superficial fluorine concentrations along the axis of a section of metallic tubing, determined through nuclear reaction $^{19}\text{F}(p, \alpha\gamma)^{16}\text{O}$

TABLE 1
ANALYSIS OF TRACES OF IMPURITIES
IN 0.5-g SAMPLES OF ALKALINE METALS (in ppm)

Metal	Carbon	Oxygen
Na (1)	0.25 ± 0.10	0.3 ± 0.1
Na (2)	4 ± 1	11 ± 3
K	6 ± 1	—
Cs	0.6 ± 0.2	6 ± 1

TABLE 2
DETECTION LIMITS FOR IRON, CHROMIUM AND NICKEL ANALYSIS
BY NEUTRONIC ACTIVATION
(Flow = 10^{12} n/cm².s¹)

Element	1 Réaction nucléaire	2 Section efficace	3 Radioisotope produit	Period	Limite de détection (µg) 4
Fe	n, γ	2.5 b	Fe 55	2.6 ans 5	5
	n, p	23 mb	Mn 54	278 j 6	10
	n, α	0.37 mb	Cr 51	27.8 j 6	100
	n, 2n	0.12 b (16 MeV)	Fe 53	9 mn 7	0.20
	n, p	0.11 b (13.5 MeV)	Mn 56	2.58 h 8	0.01
	n, γ	0.98 b	Fe 59	45 j 6	1*
	n, α	< 1.5 mb	Cr 55	3.6 mn 7	300
Cr	n, γ	13.5 b	Cr 51	27.8 j 6	0.005 **
	n, p	77.7 b (14 MeV)	V 52	5.77 mn 7	0.02
	n, γ	0.38 b	Cr 55	3.6 mn 7	0.20
Ni	n, p	0.56 b (14 MeV)	Co 58	71 j 6	0.02
	n, 2n	40.6 mb (14 MeV)	Ni 57	36 j 6	0.05
	n, np	0.16 b (14 MeV)	Co 57	267 j 6	0.20
	n, p	5 b	Co 60	5.2 ans 5	0.10
	n, p	0.18 b (14 MeV)	Co 61	1.65 h 8	0.70
	n, γ	15 b	Ni 63	125 ans 5	6
	n, α	5.7 mb	Fe 59	45 j 6	20
	n, γ	1.6 b	Ni 65	2.65 h 8	0.09

Key:

- | | |
|--------------------------|------------|
| 1. Nuclear reaction | 5. Years |
| 2. Effective section | 6. Days |
| 3. Radioisotope produced | 7. Minutes |
| 4. Detection limit (µg) | 8. Hours |

*Or 1 ppm for a one-gram sample

**Or 1 ppm for a single 5-mg sample

TABLE 3
SENSITIVITY ATTAINED BY THE GAMMA PHOTON ACTIVATION METHOD

Element	Radioisotope mesuré ¹	Period	Teneur minimale dosable ² ($\mu\text{g/g}$ ou ppm)
Carbon	^{14}C	20.3	0.02 à 0.2
Azote ³	^{14}N	9.96	0.1 à 0.3
Oxygen	^{16}O	2.03	0.04 à 0.2
Fluor ⁴	^{19}F	110	0.02 à 0.07

Key:

- | | |
|--|-------------|
| 1. Radioisotope measured | 3. Nitrogen |
| 2. Minimum measurable content
(in $\mu\text{g/g}$ or ppm) | 4. Fluorine |

Orders of magnitude of minimum measurable carbon, nitrogen, oxygen and fluorine contents by activation (γ , n) by means of slowed gamma photons produced by an electron beam with an intensity of 100 μA and maximal energy of 30 to 40 MeV. The 1-gram samples were irradiated for a period equal to the half-life of the radioisotope measured.

TABLE 4
SENSITIVITY ATTAINED BY THE CHARGED-PARTICLE ACTIVATION METHOD

Element	¹ Moyen d'activation	² Radioisotope mesuré	Period. (mn)	Teneur minimale dosable ³ (10 ⁻³ µg/g ou ppb)
Bore ⁴	⁶ ⁷ p de 10 à 15 MeV d de 10 à 20 MeV	¹¹ C	20,3	0,1 à 0,5 0,1 à 1
Carbon	d de 10 à 20 MeV	¹⁴ N	9,96	0,2 à 0,5
Azote ⁵	d de 10 à 20 MeV	¹⁵ O	2,03	0,1 à 0,5
Oxygen	¹⁶ O de 10 à 20 MeV ¹⁸ O de 34 à 42 MeV	¹⁸ F	110	0,5 à 2 0,5 à 1

Key:

- | | |
|--|-------------|
| 1. Activation method | 4. Boron |
| 2. Radioisotope measured | 5. Nitrogen |
| 3. Minimum measurable content
(in 10 ⁻³ µg/g or ppb) | 6. From |
| | 7. To |

Orders of magnitude of the minimum quantities of boron, carbon, nitrogen and oxygen measurable by means of activation by a 10-µA beam of charged particles for a period equal to the half-life of the radioisotope measured.

TABLE 5
NUCLEAR REACTIONS LEADING TO EMISSION OF CHARGED PARTICLES

1 Elément considéré	2 Réaction nucléaire mise en œuvre	3 Energie des particules incidentes (keV)	4 Limite de détection ($\mu\text{g}/\text{cm}^2$)
Lithium	${}^7\text{Li} (p, \alpha) {}^4\text{He}$	1 500	$5 \cdot 10^{-1} (*)$
Carbon	${}^{12}\text{C} (d, p) {}^{13}\text{C}$	1 000	$10^{-4} (*)$
Azote 5	${}^{14}\text{N} (d, \alpha) {}^{12}\text{C}$	1 000	$10^{-3} (**)$
Fluor 6	${}^{19}\text{F} (p, \alpha) {}^{16}\text{O}$	1 355	$5 \cdot 10^{-1} (**)$

Key:

1. Element in question
2. Nuclear reaction initiated
3. Energy of incident particles in keV
4. Detection limit ($\mu\text{g}/\text{cm}^2$)
5. Nitrogen
6. Fluorine

*Or approximately 1 ppm in a 1-micron layer

**Or approximately 40 ppm in a 3-to-4-micron layer

The above method makes it possible to detect lithium, carbon, nitrogen and fluorine in superficial layers. The detection limits are given for incident particles having the indicated energy levels. Irradiation lasts for 10 minutes.

TABLE 6
POSSIBLE ACTIVATION PROCEDURES FOR OXYGEN MEASUREMENT

Procédé d'activation 1	Teneur minimale dosable ($\mu\text{g/g}$ ou ppm) 2
$^{16}\text{O} (\text{n}, \text{p}) ^{16}\text{N}$	1
$^{16}\text{O} (\gamma, \text{n}) ^{16}\text{O}$	0.01 à 0.1
$^{16}\text{O} (\text{p}, \alpha) ^{13}\text{N}$	10^{-1}
$^{16}\text{O} (\text{p}, \text{n}) ^{18}\text{F}$	0.1
$^{16}\text{O} (\text{d}, \text{n}) ^{17}\text{F}$	10^{-1} à 10^{-1}
$^{16}\text{O} (\text{t}, \text{n}) ^{18}\text{F}$	10^{-1} à 10^{-1}
$^{16}\text{O} (^{3}\text{He}, \text{X}) ^{18}\text{F}$	10^{-1}
$^{16}\text{O} (\alpha, \text{X}) ^{18}\text{F}$	10^{-1}

Key:

1. Activation procedure 2. Minimum measurable content
(in $\mu\text{g/g}$ or ppm)

Orders of magnitude for minimum measurable contents.

TABLE 7
ANALYSIS OF AQUEOUS CORROSION PRODUCTS OF AUSTENITIC STEELS
BY NEUTRONIC ACTIVATION (in ppm)

Éléments dosés 1	Echantillon A 2	Echantillon B 3
Fe	57 ± 5	—
Ni	6.5 ± 0.7	7 ± 0.6
Cr	0.15 ± 0.01	—
Cu	0.062 ± 0.006	—
Cu	0.038 ± 0.004	0.025 ± 0.002
Ti	—	0.01 ± 0.001
U	2.1 ± 0.2	0.85 ± 0.08

Key:

1. Elements measured 3. Sample "B"
2. Sample "A"

Mass of samples was several milligrams. Nickel and copper were separated chemically.

TABLE 8
ANALYSIS OF PURE IRON BY NEUTRONIC ACTIVATION
(in ppm, in the absence of γ or p counterindications)

Impu- retés 1	Teneurs 2	Impu- retés 1	Teneurs 2
Cr	0.10 $\pm$ 0.01	P	1.2 $\pm$ 0.1
Cu	0.25 $\pm$ 0.02	Si	0.23 $\pm$ 0.10
K	$\sim$ 0.001	Zn	$\sim$ 0.006
Mn	0.115 $\pm$ 0.010	C	0.4
Mo	0.055 $\pm$ 0.006	N	0.8 γ
Na	$\sim$ 0.04 $\cdot$ 10 ⁻¹	O	0.08
Ni	0.155 $\pm$ 0.020	S	0.1 γ

Key:

1. Impurities 2. Contents

TABLE 9
ANALYSIS BY GAMMA PHOTON OR HELION-3(*) ACTIVATION (in ppm)

Metal	Carbon	Azote 1	Oxygen
Nickel	8.6	9.3	8.4
Cobalt	0.5	0.3	1.2
Silicium 2	0.05	0.1	0.1 *

Key:

1. Nitrogen 2. Silicon

TABLE 10
ANALYSIS OF REFRACTORY METALS (in ppm)

Métal	Azote 1	Oxygen	Carbon
Mo	0.5 $\pm$ 0.1 (γ) (0.3 à 13) *	7 $\pm$ 2 (γ) (9 à 11) *	1 à 2 (γ)
Ta	1.5 $\pm$ 0.2 (p)	1.6 $\pm$ 0.2 (γ) (3 à 11) *	1 à 2 (γ)
W	0.06 $\pm$ 0.01 (p) (0.1 à 5) *	1.9 $\pm$ 0.3 (γ) (1.5 à 11) *	

Key:

1. Nitrogen

*Reductive fusion, Kjeldahl or mass spectrometry.
Otherwise, γ or p activation analysis.