Fyz. čas. SAV 21 (1971), No. 4

A QUICK METHOD FOR DETERMINING THE ABSOLUTE OXYGEN CONTENT IN POWDER IRON

ARNOLD ADÁMEK,* FRANTIŠEK SEVERA,* Bratislava

The $^{16}O(n, p)^{16}N$ reaction with 14 MeV neutrons has been used for absolute oxygen content determinaticn. The results of the analyses of 40 powder iron samples are presented. Only 2–3 min. were necessary to analyze a sample.

I. INTRODUCTION

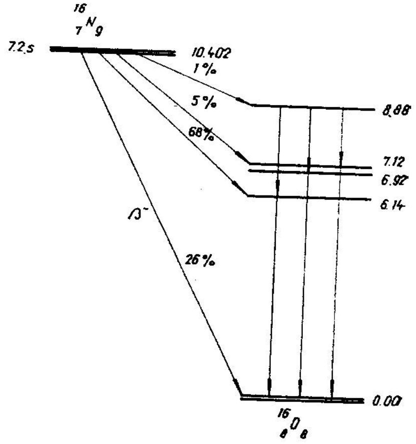
The activation analysis method using the $^{16}O(n, p)\,^{16}N$ reaction with 14 MeV neutrons seems to be a quick and nondestructive method for the oxygen content determinantions in various materials including steel, powder iron, copper etc. The fact of the half-life of ^{16}N being equal to 7·2 s [1] assures a saturation time below one minute and thus makes the use of the afore mentioned reaction very suitable for a rapid oxygen content determination. Several commercial devices for oxygen analysis in various materials have already been designed on the basis of the above reaction [2–4]. A succesful attempt to construct such a device using our available parts has been made at the Department of Nuclear Physics for commercial purposes and industrial routine analyses. The method described has been used for determining oxygen traces in semiconductor materials [5]. Results are presented of analyses of 40 powder iron samples for absolute oxygen content obtained while testing the equipment.

II. DETERMINATION OF OXYGEN BY 14 MeV DEUTRON ACTIVATION

Fig. 1. presents the decay scheme of ^{16}N from the $^{16}O(n, p)\,^{16}N$ reaction with 14 MeV energy neutrons. The method used consists of the irradiation of the sample at the target of the neutron generator and of the consequent counting of the activity rate at the scintillation detectors. Because of the short half-lif of ^{16}N, the use of a fast sample transfer system between these two positions has been found inevitable. The method of determination is relative, which

*) Fyzikálny ústav SAV, BRATISLAVA, Dúbravská cesta.

Fig. 1. Decay scheme of ^{16}N.



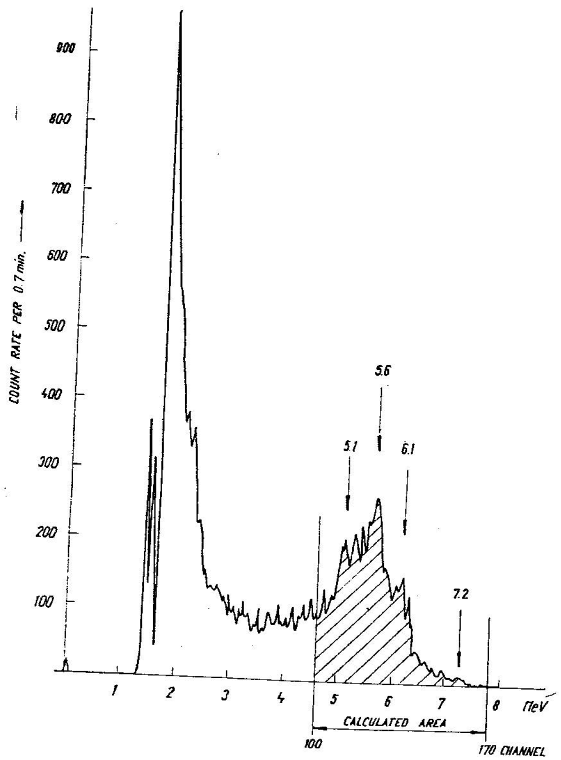


Fig. 2. Gamma-ray spectrum of ^{16}N.

means that the sample activity rate is being compared to that of the standard activated under the same conditions. Appropriate measures had to be taken to assure virtually the same conditions and also some corrections for the systematic errors should have been used. Among several different methods of internal, or external neutron flux monitoring [6] that one using a comparator enabling a reduction of the sample counting measured to the mean value of neutron flux, has been chosen. A typical spectrum of ^{16}N taken with a 3″ × 3″ NaI (Tl) scintillation detector is in Fig. 2. Only the counts registered

within the energetic interval 4·8 MeV to 7·8 MeV have been taken into consideration. A detailed discussion of the method briefly described here has been given in paper [5].

III. AN AUTOMATIC EQUIPMENT FOR THE OXYGEN DETERMINATION (OXAN) BY MEANS OF 14 MeV NEUTRON ACTIVATION

An automatic equipment for the oxygen determination (OXAN) which has been designed and constructed at the Department of Nuclear Physics consists of the following basic parts: neutron generator NG — I; dual pneumatic tube transfer system; sequence programmer; gamma-ray counting system; data output system.

The usual accelerator type, fast neutron generator NG — I, developed at the Department of Nuclear Physics [7] operating at the accelerator voltage up to 150 kV and providing the maximum total neutron yield of the order of 10^{10} n . s^{-1} has been used in the experiment. Samples were automatically transferred along the dual pneumatic transfer system [8] from the irradiation to the counting positions. An automatic control of the entire process of irradiation, transfer and counting has been provided by the sequence programmer [9].

The gamma-ray counting system consisted of two 3″ × 3″ NaI (Tl) scin-

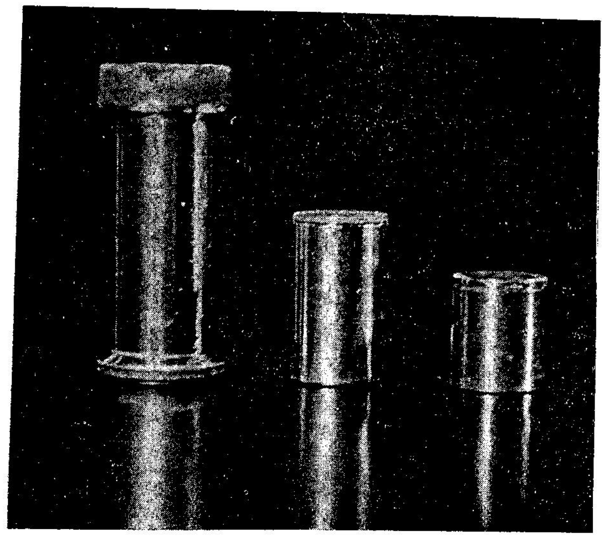


Fig. 3. OFHC-copper capsules.

Table 1

Determination of oxygen in powder iron

Sample No.	Comparator counts (background subtracted)	Sample counts (background subtracted)	Sample counts (normalized neut. flux)	Weight of sample [g]	Oxygen content mg O_2	Oxygen content % O_2
1	13 546	601	482.4	4.537	45.16	0.99 ± 0.04
2	11 103	1331	1306.6	4.448	122.04	2.74 ± 0.08
3	11 665	902	839.8	3.091	78.62	2.54 ± 0.08
4	6 023	326	578.3	3.882	54.12	1.39 ± 0.05
5	9 602	883	997.3	2.921	93.36	3.19 ± 0.09
6	14 520	809	599.4	2.659	56.11	2.11 ± 0.07
7	17 291	513	358.7	2.620	33.58	1.28 ± 0.05
8	16 413	3391	2247.6	5.906	210.41	3.56 ± 0.07
9	14 482	636	473.4	3.494	44.32	1.27 ± 0.05
10	14 331	451	341.7	3.582	31.99	0.89 ± 0.05
11	15 238	369	251.2	3.989	24.08	0.60 ± 0.04
12	11 732	351	324.6	3.314	30.39	0.92 ± 0.05
13	11 990	294	236.2	3.719	24.64	0.66 ± 0.04
14	13 942	617	476.1	4.440	44.51	1.00 ± 0.06
15	11 672	604	559.4	4.129	52.34	1.27 ± 0.07
16	12 105	2254	2023.1	5.774	189.39	3.28 ± 0.09
17	12 363	1892	1663.7	5.462	155.75	2.85 ± 0.06
18	11 137	339	322.6	4.335	30.20	0.69 ± 0.04
19	8 403	314	401.5	1.911	37.59	1.97 ± 0.12
20	10 796	308	302.5	3.795	28.32	0.75 ± 0.04
21	11 037	3080	3038.1	5.597	284.41	5.08 ± 0.10
22	3 452	131	410.0	3.763	38.38	1.02 ± 0.10
23	11 061	446	432.8	3.694	40.52	1.09 ± 0.05
24	9 304	283	327.6	3.513	30.67	0.87 ± 0.05
25	11 113	564	547.9	3.648	51.29	1.41 ± 0.05
26	12 081	514	513.3	3.967	48.05	1.21 ± 0.05
27	9 482	673	773.0	3.401	72.36	1.95 ± 0.08
28	7 881	427	519.0	3.453	54.20	1.57 ± 0.07
29	9 906	2073	2274.0	3.054	212.88	6.79 ± 0.14
30	7 553	262	374.6	3.369	35.07	1.04 ± 0.09
31	10 437	354	375.5	2.872	35.15	1.22 ± 0.07
32	9 873	658	723.1	3.322	67.69	2.04 ± 0.08
33	11 724	443	408.5	3.147	38.24	1.22 ± 0.06
34	11 823	473	432.6	3.467	40.50	1.17 ± 0.06
35	8 887	493	600.3	3.515	56.20	1.60 ± 0.06
36	5 481	211	416.9	3.231	39.03	1.21 ± 0.09
37	9 493	427	481.5	3.965	45.07	1.14 ± 0.06
38	7 932	238	315.5	2.854	39.53	1.03 ± 0.09
39	7 213	881	1327.9	4.951	124.31	2.50 ± 0.10
40	11 603	382	356.4	3.117	33.36	1.07 ± 0.05

tillation detectors TESLA – Liberec and pulses were fed into the multichannel analyser Victoreen Tullamore U.S.A. The use of a multichannel analyser is not inevitable: the system of commercial discriminators and scalers (TESLA) is quite a sufficient counting device for purposes of analysis. Either a manual or a computer evaluation can be selected. For the computer evaluation a parallel converter and punch-tape output is available. The program was written in FORTRAN IV language.

IV. RESULTS

Powder iron samples were carries in OFHC-copper ($< 10^{-2}$ % O_2) capsules so that the blank value affecting the accuracy of measurement [10] was minimized (Fig. 3). Samples were irradiated for 30 s and counted for 42 s. The background was subtracted before the number of counts measured had been reduced to the normalized (main value) of the neutron flux. Corrections were made for the blank value and for the residual oxygen content in the empty capsule. Several standards ($C_4H_6O_6$, Al_2O_3, $C_2H_2O_4$) were used and no significant differences between the results were found. The calculations were carried out by the CDC-3300 computer. The results are given in Table 1. The experimental errors did not differ significantly from the statistical errors of counting. The analysis time per sample was approximately 2–3 min.

V. CONCLUSION

The fast neutron activation method has proved to be a rapid method for the determination of oxygen in powder iron. Short saturation time and fairly good sensitivity (of the order of 10^{-2} %) have been found to be the main advantages of the method. The sensitivity of the method could be further highly improved if a neutron generator with a greater total neutron yield and a better OFHC-copper for the capsules were used.

The authors are indebted to Mr. J. Stein for working out the program for the computer evaluation of measurements and wish to thank Mr. J. Král for his technical assistance.

The authors gratefully wish to thank for the use of the neutron generator developed by Mr. Š. Bederka.

REFERENCES

[1] Faull M., Nucl. Sci. Abstr. *11* (1957), ref. 4707.
[2] Nargolwalla S. S., Crambes M. R., De Voe., Anal. Chem. *40* (1968), 666.
[3] *Neutrinox-automatic installation for the determination of oxygen in steel by neutron activation analysis.* SAMES, 21, rue Jean-Macé, 38 Grenoble, France.

[4] Ворзатз Б., Сабо Е., Научно-техническая конференция для активационного анализа. Сборник докладов, Москва 1969.
[5] Adámek A., Severa F., *Stanovenie stôp kyslíka v polovodičových materiáloch pomocou aktivačnej analýzy*. Report of the Institute of Physics of the Slovak Academy of Sciences No. 34/69, Bratislava 1969.
[6] Mott W. E., Orange J. M., Anal Chem. *3* (1965), 1338.
[7] Bederka Š., Dissertation. Bratislava 1967.
[8] Adámek A., Severa F., J. Radioanal. Chem., to be published.
[9] Adámek A., Severa F., Czech. J. Phys. *B 20* (1970), No. 11.
[10] Nargolwalla S. S., Przybylowicz E. P., Suddueth J. E., Birkhead S. L., Anal. Chem. *41* (1969), 168.

Received March 24th, 1970