Determination of Sunflower Seed Oil Content Using Natural Sunflower Oil: Calibration of a Pulsed NMR Analyzer

Oleg S. Agafonov and Sergey M. Prudnikov

Abstract The present study substantiates the application of natural sunflower oil for the calibration of pulsed NMR analyzers, as well as for the identification and quality assessment of oilseeds and their products using the NMR method. Being the essential characteristic of sunflower seeds, the oil content is mandatory for the certification of agricultural products and their cost assessment. The first calibration option includes the measurement of oil NMR characteristics with the oil content determined using the Soxhlet exhaustive extraction. The second option involves the calibration of a NMR analyzer using sunflower oil, obtained by pressing identical cultivars and hybrids, as well as that acquired at a retail network. A comparison of the obtained calibration dependencies showed their similar nature. The application of sunflower oil for the calibration of NMR analyzers provides no increase in the oil content measurement error as compared to the first calibration option. Therefore, using natural oil for the calibration of NMR analyzers can considerably simplify the calibration process, reduce the calibration duration from 3–4 days to 3–4 h, as well as to exclude toxic solvents and additional high-value equipment from the process without essential variations in the oil content measurement error.

Keywords NMR analyzer · Calibration · Oil content · Moisture content · Reference materials · Sunflower seeds · Sunflower oil

Abbreviations

RM	Reference material
TAG	Triacylglycerol
NMR spectroscopy	Nuclear magnetic resonance spectroscopy

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Introduction

In accordance with GOST 22391-2015 [1], the oil-and-fat industry identifies the seed oil content as the main quality indicator of raw materials during their gathering and processing. The oil content comprises the content of raw fat and accompanying lipoid substances, which, together with fat, pass into the ether extract from the seeds. In accordance with GOST 10857-64 [2], the determination of this indicator is based on the method of Soxhlet exhaustive extraction. A drawback of this method consists in its duration, application of toxic chemical solvents, low productivity, and high requirements to the personnel qualification.

At present, due to the wide development of technical equipment and information technologies, instrumental methods, such as IR spectroscopy and pulsed NMR methods, which can be used to obtain information about the seed oil content, are quite common. The determination of oil content using the method of IR spectroscopy is typically performed in accordance with GOST 32749-2014 [3]. In the case of sunflower seeds, this method is unpopular due to significant errors (up to 2 abs%) and the dependence of measurement results on the seed appearance and their fineness degree. In addition, this method requires the calibration of IR analyzers using a large number of seeds with a certain value of determined indicators obtained using referee methods [4–8].

In the contemporary Russian oil-and-fat industry, the most widespread instrumental method for determining the oil content of seeds and products of their processing involves pulsed nuclear magnetic resonance (NMR). This is explained by its analytical simplicity, lack of a complex sample preparation, high accuracy (error of not more than 0.6 abs%, which is comparable in accuracy with a referee chemical method), promptness (the analysis of one sample takes < 30 s), lack of the effect caused by the operator's subjectiveness on the analytical results, as well as ease of implementation. Currently, AMV-1006M (V.S. Pustovoit All-Russian Research Institute of Oil Crops, VNIIMK, Russia) is the most common NMR analyzer used at Russian enterprises. The determination of oil content using this instrument is based on the substantiated scientific and methodological approach with the rank of 0.04 [9].

The calibration of operating NMR analyzers for the determination of oil content in sunflower seeds is carried out using GSO 3107-84[1] state reference materials [10, 11], which were used for the verification of AMV-1006M NMR analyzers until the expiration of their Type Approval Certificate in 2016.

These reference materials (RM) are made on the basis of chemically inert substances-imitators, thermally resistant, resistant to thermal oxidation and ultraviolet effects, as well as having the dielectric properties (of organosilicon liquids) [12–14] of oil NM relaxation characteristics.

[1] GSO 3107-84 Reference materials of the approved type of oil content and moisture content of sunflower seeds (set). Available via FIF EUM. www.fgis.gost.ru/fundmetrology/registry/19/items/393823. Accessed 12 September 2022 (In Russ.).

Imitated values of indicated GSO 3107-84 were assigned using an NMR analyzer, whose calibration and testing were performed using sunflower seeds with the oil content determined by the Soxhlet method of exhaustive extraction. The drawbacks of this calibration method include its duration, high requirements to the operator's qualification, as well as the application of specially prepared traceable seeds. It should be noted that even specially prepared seeds have a scattering in the measured oil content values in individual samples, isolated from one specimen using the method of exhaustive extraction to 1 abs%. This is explained by the fact that seeds represent a natural heterogeneous object [15, 16].

Let us note that one of the key features of the NMR method comprises a functional dependence between the oil content in the analyzed seed sample and the amplitude of the NMR signal obtained from the protons of triacylglycerins (TAG) contained in the oil.

At present, the NMR method is widely used for determining the oil content of seeds and oil-containing raw materials [17–24],[2] which is explained by its application simplicity, high metrological characteristics, sample preparation simplicity, ecological safety, as well as the non-destructive nature of analysis. More than 350 enterprises of the Russian oil-and-fat industry currently use AMV-1006M NMR analyzers to quickly obtain information on the oil content at all stages of gathering, storage, and processing of oil seeds.

At the same time, the calibration of quantitative NMR analyzers represents a complex problem.

Currently, several main methods of calibrating quantitative analyzers, used for the quality assessment of lipid-containing raw materials, can be found in literature:

- using natural samples[3,4];
- using imitator RMs produced from chemically inert substances [13, 14, 24];
- using samples, obtained on the basis of natural components, e.g., oil cakes and oil (see footnote 3) [25].

In this study, we aim to scientifically and experimentally substantiate the application of sunflower oil samples in the calibration of NMR analyzers.

[2] The minispec Oil content and moisture in seeds and nuts. Available via: URL: http://spectrante.ru/images/pdf_series/Zernovie.pdf. Accessed 12 September 2022 (In Russ.).

[3] Measurement of oil component in dried palm mesocarp. Available via: Oxford Instruments. https://nmr.oxinst.com/assets/uploads/18_Measurement_of_Oil_Content_in_Dried_Palm_Mesocarp.pdf. Accessed 12 September 2022.

[4] Measurement of oil and water in seeds according to ISO 10565. Available via Oxford Instruments. https://nmr.oxinst.com/assets/uploads/3_3344_MR_Oilseeds_App%20Note_Web.pdf. Accessed 12 September 2022.

Materials and Methods

Experiments were carried out at the Central Experimental Base of V.S. Pustovoit All-Russian Research Institute of Oil Crops (Krasnodar) in 2021–2022. Traceable sunflower seed samples of VNIIMK[5] breeding and two samples of commercially distributed oil were prepared, including the refined and deodorized oil of the "BLAGO" trademark (Russia) and non-refined oil of the "STAVROPOL'E" trademark (Russia), acquired in a retail network.

Sunflower samples were cleaned off damaged seeds and waste admixtures. The oil and moisture content of seeds was preliminarily determined using an AMV-1006M NMR analyzer in accordance with GOST 8.597-2010 [26]. The seed samples presented in the study belong to contemporary high-productive varieties. The acid number of the seed oil was determined according to GOST 31933-2012 [24, 27] using the titrimetric method with visual indication.

The determination of the oil content in seeds was carried out by the method of exhaustive extraction in four repetitions for each sample in order to reduce the measurement error, in accordance with the procedure developed based on GOST 10857-64. The duration of extraction was 24 h, at a temperature providing a number of siphonings from 7 to 10 per hour. The completeness of extraction was checked using a watch glass sample. The average value of four measurements was taken as the final result.

The oil from sunflower seeds was obtained using a Laboratoroff PR-L laboratory hand press (LLC Eltemix, Russia) with a force of 12 tons. The obtained oil was further filtered to remove seed particles, trapped in the oil during its pressing, using the FS laboratory filtering paper.

In order to construct the oil content calibration curves, from each oil sample, both obtained by pressing and acquired at a retail network, five weighments evenly distributed in the range from 2000 to 7000 g with the accuracy of 0.001 g were taken using an AND HK-50AG laboratory weights (AND, Japan).

Prior to measuring the oil proton NMR signals, the prepared samples were thermostated at a temperature of 23 ± 0.5 °C for 2 h in a TVL-K(50)B thermostat (CJSC INSOVT, Russia). The thermostating of samples comprises an important stage, since the temperature has the essential effect on the NM relaxation characteristic of oil samples [21].

[5] Sunflower varieties of VNIIMK breeding, Russia:

UMNIK, oil content 30–35%. Available via VNIIMK. https://vniimk.ru/products/belosnezhnyy-sq/. Accessed 12 September 2022 (In Russ.).

DZhIN, oil content 44–46%. Available via VNIIMK. https://vniimk.ru/products/dzhinn/. Accessed 12 September 2022 (In Russ.).

IMIDZh, oil content 48%. Available via VNIIMK. https://vniimk.ru/about/fgup/Каталог%20ВНИИМК.pdf. Accessed 12 September 2022 (In Russ.).

SPK, oil content 46–47%. Available via VNIIMK. https://vniimk.ru/products/spk/. Accessed 12 September 2022 (In Russ.).

BELOSNEZhNYI, oil content 30–35%. Available via VNIIMK. https://vniimk.ru/products/belosnezhnyy-sq/. Accessed 12 September 2022 (In Russ.).

Measurements were conducted using typical AMV-1006M NMR analyzers of the oil and moisture content (VNIIMK, Russia). In accordance with the analytical procedure, when performing measurements by an AMV-1006M NMR analyzer, the volume of each analyzed seed weighment is equal to 25 cm^3.

For the control of NMR analyzers and primary processing of NMR signals, obtained using the studied oil samples, the own software was used [28]. The obtained data were statistically analyzed using Statistica and Excel software.

Results and Discussion

The main characteristics of sunflower seeds used in the study are presented in Table 1.

It is known [2] that, for oilseeds with a moisture content of < 8%, the intensity of NM relaxation characteristics is determined exclusively by the protons of the oil and accompanying substances. This is due to the high degree of connection between water molecules and the protein part of the seeds. The data in Table 1 show that the moisture content of the samples under study is much lower. Therefore, the resulting analytical parameter will further characterize only TAG protons.

The seed samples prepared for the analysis characterize the range of the oil content from 32.1 to 56.3%. The seeds are healthy; their acid number corresponds to the first-class sunflower and does not exceed 0.8 mg KOH/g for all samples.

At the first stage of the study, we obtained the NM relaxation characteristics of the prepared oilseeds and determined their oil content using the Soxhlet method of exhaustive extraction. The obtained calibration curve, describing the dependence of the oil mass on the amplitude of the NMR signal, is shown in Fig. 1.

The resulting curve is characterized by a high correlation coefficient of 0.9999. The main drawback of the described calibration technique is the complexity of implementation, duration, and high requirements for the qualification of personnel.

Table 2 shows the calculated values of the oil mass in sunflower seeds and the actual values obtained by the extraction method.

Table 2 shows that the maximum error in measuring the oil mass in the analyzed sample by the NMR method does not exceed 28 mg, which, in terms of the oil

Table 1 Characteristics of the studied sunflower seeds

Seed sample	Indicators		
	Oil content, %	Moisture content, %	Oil acid number, mg KOH/g
BELOSNEZhNYI	32.1	6.9	0.6
IMIDZh	41.3	5.2	0.7
DZhIN	42.5	5.5	0.6
SPK	48.2	5.3	0.8
UMNIK	56.3	4.8	0.6

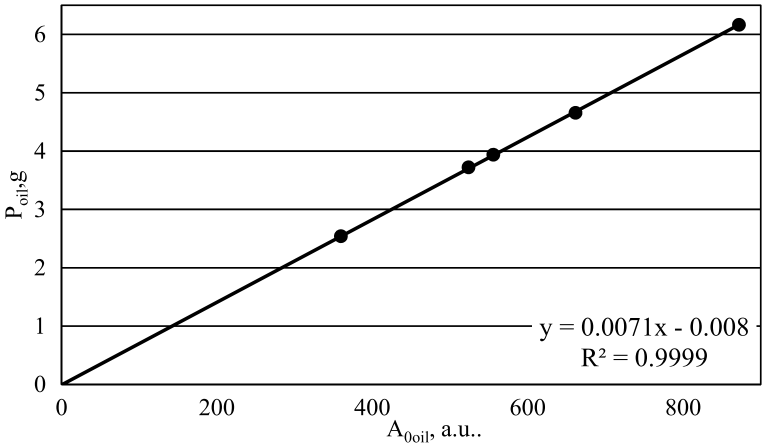

Fig. 1 Dependence of the mass of the sunflower oil, obtained by the Soxhlet extraction, on the amplitude of the NMR signal

Table 2 Calculated and actual (obtained by the extraction method) values of the oil mass in sunflower seeds

Seed sample	Oil mass in the analyzed sample (P_{oil}), g		Deviation from the actual value, g	Deviation in terms of the oil content, %
	Actual	Calculated		
1	2.540	2.539	0.001	0.02
2	3.719	3.706	0.013	0.15
3	3.936	3.932	0.004	0.04
4	4.656	4.684	– 0.028	– 0.29
5	6.164	6.178	– 0.014	– 0.13

content, taking into account the seed moisture content and the mass of the sample with a volume of 25 cm^3, is < 0.29%.

At the next stage, the possibility of using oil, obtained by pressing sunflower seeds, for the calibration of an NMR analyzer was investigated. Figure 2 illustrates the dependence of the oil mass on the amplitude of the NMR signal obtained using the oil pressed from sunflower seeds of three contemporary high-oil varieties (Dzhin, Imidzh, SPK).

According to the presented curve, the variety special features of seeds cause no significant effect on the amplitude of the oil NMR signal and, consequently, on the obtained analytical dependence. The difference between the obtained curve and that shown in Fig. 1 can be explained by the introduction of different amounts of accompanying substances in the process of oil extraction and pressing.

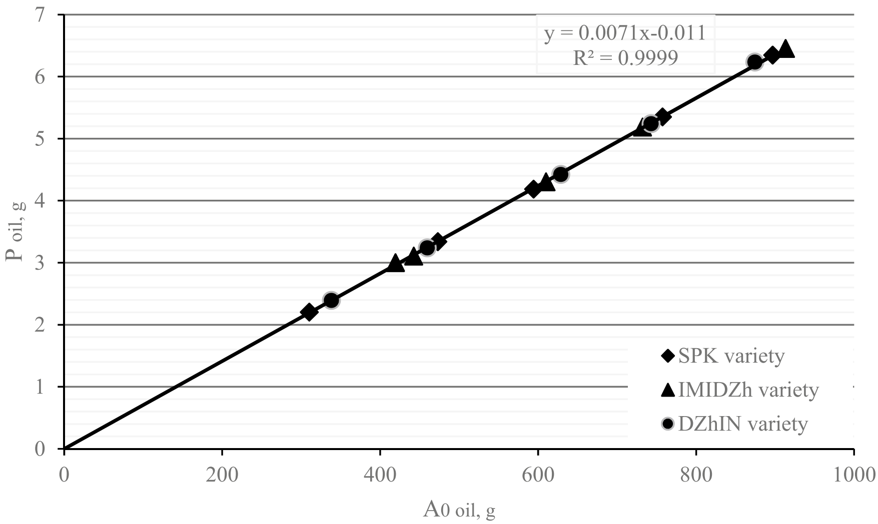

Fig. 2 Dependence of the pressed oil mass on the amplitude of the proton NMR signal

Table 3 provides the calculated values of the oil mass in sunflower seeds according to the calibration of an NMR analyzer using pressed oil.

The application of a calibration curve, obtained using pressed oil, leads to a maximum measurement error of not more than 32 mg or 0.33 abs% in terms of the oil content taking into account the seed moisture content and the mass of the sample with a volume 25 of cm^3 in the entire studied range.

At the third stage of the study, two samples of sunflower oil were used: refined and deodorized of the "BLAGO" trademark and non-refined of the "STAVROPOL'E" trademark, acquired at a retail network.

Figure 3 plots the dependence of the oil mass on the amplitude of the NMR signal, obtained using oil acquired at a retail network.

Table 3 Calculated values of the oil mass in sunflower seeds according to the calibration of an NMR analyzer using pressed oil

Seed sample	Oil mass in the analyzed sample (P_{oil}), g		Deviation from the actual value, g	Deviation in terms of the seed oil content, %
	Actual	Calculated		
1	2.540	2.543	– 0.003	– 0.03
2	3.719	3.710	0.009	0.10
3	3.936	3.936	0.000	0.00
4	4.656	4.688	– 0.032	– 0.33
5	6.164	6.182	– 0.018	– 0.16

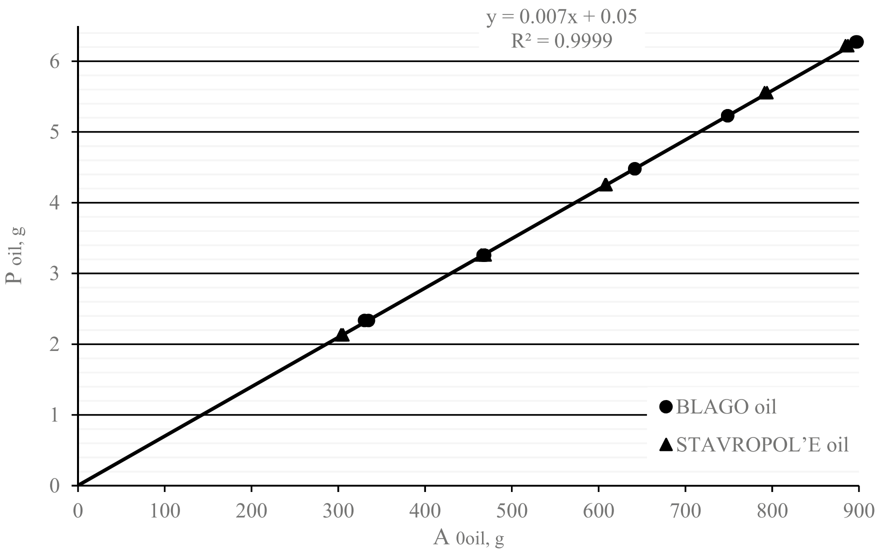

Fig. 3 Dependence of the commercially distributed oil mass on the amplitude of the proton NMR signal

As in the case of pressed oil, the obtained curve of the dependence between the mass of the analyzed oil sample and the amplitude of the oil NMR signal has a linear nature with the high correlation coefficient.

Table 4 provides the calculated values of the oil mass in sunflower seeds according to the calibration of the NMR analyzer using commercially distributed oil.

The application of a calibration curve, obtained using commercially distributed oil, leads to a maximum measurement error of not more than 28 mg or 0.35 abs% in terms of the oil content taking into account the seed moisture content and mass of the sample with a volume 25 of cm^3 in the entire studied range.

Table 4 Results of the oil mass measurement in sunflower seeds according to the calibration, obtained using commercially distributed oil

Seed sample	Oil mass in the analyzed sample (P_{oil}), g		Deviation from the actual value, g	Deviation in terms of the seed oil content, %
	Actual	Calculated		
1	2.540	2.568	– 0.028	– 0.35
2	3.719	3.719	0.000	0.00
3	3.936	3.942	– 0.006	– 0.06
4	4.656	4.683	– 0.027	– 0.28
5	6.164	6.155	0.008	0.08

At the next stage, the oil samples were studied for the temporal stability of NM relaxation characteristics. Three weighments of both refined deodorized and pressed oil were selected for a storage at a temperature of 8 °C in tightly-closed glass cups. Once a week, the samples were taken out of the refrigerator and thermostated at a temperature of 23 ± 0.5 °C for 4 h followed by threefold measurements of the oil NMR signal amplitude. The mean of three measurements was taken as the measurement result. Further, using the obtained earlier calibration equations, the oil masses of the studied samples were calculated and the deviations from the basic value were determined (Table 5).

According to Table 5, the NM relaxation characteristics of the refined oil and, consequently, the calculated values of the oil mass remain stable during the considered storage time. However, for the pressed oil samples, the measured amplitude of the NMR signal of TAG protons considerably altered and led to a decrease in the calculated oil mass value by 570 mg.

Thus, according to the results of the temporal stability study, pressed oil samples can preserve their proton NM relaxation characteristics during not more than 5–7 days, while the refined deodorized samples demonstrate the stability during not < 3 months at a temperature of 8 °C. This can be explained by the presence of a large quantity of free radicals, moisture, and other accompanying substances in pressed oil, facilitating the flow of oxidation processes, which lead to an increase in the quantity of free fatty acids and other products of oxidizing reactions.

Table 5 Results of the oil mass measurement in the analyzed samples depending on the storage time

Week	Deviation of measured oil mass values, g For the samples					
	Refined deodorized			Pressed		
	1	2	3	1	2	3
0	0.000	0.000	0.000	0.000	0.000	0.000
1	– 0.012	– 0.007	– 0.012	0.048	0.049	0.051
2	0.009	– 0.011	–0.010	0.046	0.051	0.052
3	– 0.012	0.003	– 0.013	0.080	0.084	0.089
4	0.011	–0.010	0.013	0.117	0.117	0.126
5	0.008	0.003	0.011	0.166	0.168	0.173
6	– 0.010	– 0.008	– 0.012	0.214	0.219	0.222
7	– 0.013	– 0.010	0.007	0.264	0.269	0.270
8	0.012	0.009	0.015	0.315	0.317	0.321
9	– 0.011	– 0.009	– 0.007	0.366	0.364	0.368
10	– 0.006	– 0.010	– 0.009	0.427	0.428	0.429
11	0.010	0.012	– 0.003	0.491	0.490	0.491
12	– 0.011	– 0.010	0.010	0.568	0.567	0.566

Table 6 Comparative characteristics of NMR-analyzer calibration methods for determining the oil content of sunflower seeds

Indicator	Calibration procedure		
Used materials	Traceable sunflower seeds	Pressed sunflower oil	Refined sunflower oil
Auxiliary equipment	Scales (Accuracy class—high-II), thermostat, Soxhlet extraction equipment	Scales (Accuracy class—high-II), thermostat, laboratory press	Scales (Accuracy class—high-II), thermostat
Calibration time	3 days	1 day	4 h
Personnel qualification requirements	High	Medium	Medium
Sample reusability	None	Can be reused within 3–5 days	Can be reused within 3 months

Comparative characteristics of the studied methods for calibrating an AMV-1006M NMR analyzer are presented in Table 6.

Conclusion

The conducted research shows that the use of sunflower oil, obtained in different ways, for the calibration of NMR analyzers can significantly facilitate the calibration process, reduce the calibration time from 3–4 days to 3–4 h, as well as to eliminate the use of toxic solvents and additional expensive equipment from the process without significantly increasing the error in determining the oil content of sunflower seeds by the NMR method.

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Author cotributions The authors have contributed equally.

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