

## COMPARISON OF THE QUALITY OF COLD PRESSED AND VIRGIN RAPESEED OILS WITH INDUSTRIALLY OBTAINED OILS

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Key words: cold pressed oil, rapeseed oil, virgin oil, fully refined oil, fatty acid composition, tocopherols, oxidative stability

This work evaluates the quality of cold pressed rapeseed oils as well as virgin oils (from seeds heated before pressing) obtained under laboratory conditions and compares them with the quality of industrially obtained hot pressed, crude, bleached and deodorised oils. The method of obtaining oils did not affect the fatty acid composition. The composition of fatty acids in all oils was typical of the low-erucic rapeseed. The analysed oils did not contain *trans* fatty acid isomers, with the exception of the deodorised oils after full refining (linoleic and linolenic 1.1%). It was found that the quality of the analysed oils depended significantly on the quality of the method of their production and further processing. The quality of cold pressed and virgin oils was good and in keeping with the expectations for edible oils. Cold pressed oils differed statistically significantly from both the virgin type oils and deodorised ones obtained from the same seeds in all the tested parameters: colour, acid and peroxide value, Totox, induction time and only in the case of anisidine value they showed a significantly lower value. Cold pressed oils (5.08 h) were less stable in Rancimat test in comparison with oils after full refining (5.37 h). The highest content of tocopherols was characteristic for crude oils (58.4 mg/100g). The refining process caused a decrease by over 40% of tocopherol content in fully refined oils. A higher by 25% content of tocopherols and the lack of *trans* fatty acid isomers speaks for the cold pressed oils.

### INTRODUCTION

Edible oils can be obtained by various methods. Using traditional technology based on pressing, then extraction of oil from raw material and later chemical refining is very energy consuming, costly and moreover it pollutes the environment [Dijkstra, 1999]. A more profitable way is the use of physical refining of the pressed oil [Cvengroš, 1995; Čmolik *et al.*, 2000]. However, the alternative, which is the most environmentally friendly may be the technology of cold pressing. This method is uncomplicated, cheap and environmentally friendly. The important limitations of this ecological method are low effectiveness of this process and problems with obtaining a constant quality oil, depending on the quality of raw material. The effectiveness of pressing can be significantly increased by applying heating to the seeds before or during pressing. However, the product is not the cold pressed oil but the virgin oil. According to the definition presented by the FAO/WHO Codex Alimentarius for virgin oils it is acceptable to introduce temperature into the technological process, contrary to cold pressed oils [Codex Alimentarius, 2001].

Cold pressed oils generally exceed refined oils in their nutritional value. They contain more natural beneficial ingredients such as tocopherols, sterols, carotenoids, and phospholipids which are partially removed as a result of oil refining [Prior *et al.*, 1991a,b; Gogolewski *et al.*, 2000; Koski *et al.*, 2003]. Many different data confirm good sensory

and chemical quality of cold pressed rapeseed oils [Rotkiewicz *et al.*, 1995; De Panfilis *et al.*, 1998; Krygier *et al.*, 1998; Rotkiewicz & Konopka, 1998; Koski *et al.*, 2002; Górecka *et al.*, 2003; Matthäus & Brühl, 2003]. The objection connected with the presence of dangerous quantities of metals ions, pesticides, polynuclear aromatic hydrocarbons in these oils has not been confirmed so far [Jankowski *et al.*, 1998; Jankowski & Obiedziński, 2000]. These oils – as natural products – are of a growing interest among consumers, however, they are still marginal on the oils market in many countries. The dominant group of edible oils are, especially in Central and Northern Europe, refined oils. Thus, it is interesting to compare edible not refined cold pressed and virgin oils and fully refined oils.

In particular, the aim of this work was the quality evaluation of the following oils: cold pressed and virgin obtained under laboratory conditions, and industrial oils: hot pressed, crude, bleached and deodorised.

### MATERIAL AND METHODS

The research material was typical industrial rapeseed of double zero variety and following oils: oils obtained in the laboratory – cold pressed oils (oil at room temperature) and virgin oils pressed from preheated seeds (100°C, 40 min). Crushed industrial rapeseed were subjected to the pressing process in the hydraulic press Caver Laboratory Press (USA)

under constant pressure of 40 MPa for 20 min; and oils from the industrial technological line (from one Polish oil mill fitted with typical equipment for oil extracting): hot pressed oils – from flaked and preheated seeds after filtration; crude oils (hot pressed + solvent extracted oils – industrial sample) before acid degumming; bleached oils – after filter press; deodorised oils – after cooling. The experiments were carried out in 6 series for each oil. Each set came from the same group of seeds and from the same production cycle, every 6 hours. Samples were frozen.

Seeds were examined according to Polish Standards for: (1) fat content by Soxhlet method according to PN-EN ISO 734-1:2000, (2) moisture by drying at 103°C for 3 h according to PN- 73/A-86912 and (3) physical contaminants according to PN-91/R-66160.

In each oil sample the following parameters were determined. Acid value (AV) expressed in mg KOH per g of oil was determined according to PN-ISO 660:1998. Peroxide value (PV) expressed in milliequivalents of active oxygen per kg of oil was determined according to PN-ISO 3960:1996. Anisidine value (AV) was determined according to PN-EN ISO 6885:2001. Totox index was calculated according to PN-93/A-86926, where Totox = 2PV + AV. Total spectrophotometric colour was determined according to PN-A-86934, where total colour = 1000 ( $A_{442} + A_{668}$ ),  $A_{442}$  – absorbance at  $\lambda = 442$  nm, oil samples were dissolved in n-hexane (1:10) and analysed as carotenoid pigments, and  $A_{668}$  – absorbance at  $\lambda = 668$  nm (oil in n-hexane (1:1)) – as chlorophyll pigments at glass absorption cell (10 mm) in UV-VIS spectrophotometer Helios  $\beta$  (Unicam). Chlorophyll pigments as pheophytin a were determined with the AOCS method [AOCS Cc 13i-96:1997]. Induction time was determined with Rancimat test in 120°C according to PN-ISO 6886:1997.

Fatty acid composition was determined with gas liquid chromatographic (GLC) method of fatty acids methyl esters according to PN-EN ISO 5509 and PN-EN ISO 5508. For GLC analysis a Hewlett-Packard 5890 Series II chromatograph equipped with a flame-ionization detector (FID) and capillary column BPX 70 (60 m x 0.25 mm) was used. The temperature program was 140-210°C. The injection and detector temperatures were 210 and 250°C, respectively. Helium was used as carrier gas.

Tocopherols were determined with the reversed phase high performance liquid chromatography (RP-HPLC) according to ISO 9936:1997. The oils were dissolved in methanol. Tocopherols were separated on Supelcosil LC-18 column (25 cm x 4.6 mm x 5  $\mu$ m). Waters chromatograph equipped with a 20  $\mu$ L flow cell and with UV-VIS detector ( $\lambda = 295$  nm) were used for the HPLC analysis. Methanol and deionized water were used (98:2) at a flow rate of 2 mL/min. Recoveries of tocopherol isomers were determined by adding known amounts of tocopherol (MERCK) to oil sample and by applying the same experimental conditions.

The obtained results were analysed statistically by means of computer program STATGRAPHICS PLUS 4.1. Means and standard deviations (SD) were calculated. One-way analysis of variance (ANOVA) was used to check the significance of statistical differences between the means at  $p = 0.05$ .

## RESULTS AND DISCUSSION

The quality of the not refined oils depends significantly on the quality of seeds and thus before the beginning of the technological process the raw material was examined. The fat content was 41% and moisture 6.2%, on average (Table 1). Thus, seeds were characterised by a typical fat content and optimal moisture necessary for safe storage of seeds without a significant change in their quality, both technological and microbiological.

For all the examined oils the composition of fatty acids was typical and characteristic for the low-erucic rapeseed oil [Codex Alimentarius, 2001]. The method of oil production did not change its composition of particular fatty acids (Table 2). Cold pressed oils, virgin type oils and hot pressed oils, crude and bleached ones did not contained harmful to human health *trans* isomers of fatty acids, however, there were 1.1% of them in the deodorised oils. Partly geometric isomerization of polyunsaturated acids (linoleic acid 0.1% and linolenic acid 1%) is caused by the process of deodorization and especially high temperature of up to 240°C during that process [Wagner *et al.*, 2000]. The more strict are the deodorization parameters

TABLE 1. Characteristics of the examined rapeseed (6 series).

Characteristic	Range	Average	SD*
Moisture (%)	6.0-6.4	6.20	0.17
Fat (%)	39.3-42.0	41.00	0.94
Physical contaminants total (%)	1.60-3.5	2.24	0.61
useful	0.8-2.0	1.14	0.39
unuseful	0.8-1.5	1.10	0.23

\*SD – standard deviation

TABLE 2. Fatty acid composition (%) of the examined oils (6 series).

Fatty acids (%)	Cold-pressed	Hot-pressed	Crude	Bleached	Deodorized
C 16:0	4.6 <sup>a</sup>	4.6 <sup>a</sup>	4.8 <sup>b</sup>	4.6 <sup>a</sup>	4.7 <sup>ab</sup>
C 16:1	0.2 <sup>a</sup>	0.3 <sup>a</sup>	0.2 <sup>a</sup>	0.3 <sup>a</sup>	0.3 <sup>a</sup>
C 18:0	1.7 <sup>a</sup>	1.7 <sup>a</sup>	1.7 <sup>a</sup>	1.7 <sup>a</sup>	1.7 <sup>a</sup>
C 18:1 (9)	56.2 <sup>a</sup>	55.8 <sup>b</sup>	55.2 <sup>c</sup>	55.7 <sup>b</sup>	55.5 <sup>c</sup>
C 18:1 (11)	3.5 <sup>a</sup>	3.1 <sup>b</sup>	3.6 <sup>c</sup>	3.5 <sup>a</sup>	3.0 <sup>b</sup>
C 18:2 <i>cis</i>	18.7 <sup>a</sup>	19.3 <sup>b</sup>	19.4 <sup>b</sup>	19.3 <sup>b</sup>	19.9 <sup>c</sup>
C 18:2 <i>trans</i>	0	0	0	0	0.1 <sup>a</sup>
C 18:3 <i>cis</i>	8.6 <sup>a</sup>	8.8 <sup>b</sup>	8.5 <sup>c</sup>	8.3 <sup>d</sup>	7.7 <sup>e</sup>
C 18:3 <i>trans</i>	0	0	0	0	1.0 <sup>a</sup>
C 20:0	0.6 <sup>a</sup>	0.6 <sup>a</sup>	0.6 <sup>a</sup>	0.6 <sup>a</sup>	0.6 <sup>a</sup>
C 20:1	2.4 <sup>a</sup>	2.4 <sup>a</sup>	2.4 <sup>a</sup>	2.4 <sup>a</sup>	2.3 <sup>b</sup>
C 20:2	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>
C 22:0	0.3 <sup>a</sup>	0.3 <sup>a</sup>	0.3 <sup>a</sup>	0.3 <sup>a</sup>	0.3 <sup>a</sup>
C 22:1	2.3 <sup>a</sup>	2.1 <sup>b</sup>	2.1 <sup>b</sup>	2.0 <sup>c</sup>	1.9 <sup>d</sup>
C 24:0	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>
C 24:1	0.1 <sup>a</sup>	0.2 <sup>b</sup>	0.2 <sup>b</sup>	0.2 <sup>b</sup>	0.2 <sup>b</sup>

\* – values marked with the same letter in a row are not significantly different at  $p = 0.05$ .

(higher temperature, longer time) the higher is the content of *trans* isomers [Płatek & Krygier, 1998]. The characteristic feature of rapeseed oils is the presence of erucic acid C22:1. Its content in the analysed oils varied from 1.9 to 2.3%.

### Comparison of laboratory cold pressed and virgin oils and industrial hot pressed oils

In respect of hydrolytic and oxidative values, cold pressed oils and virgin oils showed a very good quality for such products: their acid value accounted for 1.92 and 2.68 mg KOH/g oil, respectively (norm up to 4), and peroxide value (PV) for respectively 1.98 and 3.11 meq/kg oil (norm up to 15) (Table 3). These values fulfil the Codex Alimentarius [2001] for these types of edible oils and were similar to those obtained in other investigations [Rotkiewicz *et al.*, 1995; Rodkiewicz & Konopka, 1998; Koski *et al.*, 2002; Górecka *et al.*, 2003].

Cold pressed oils differed significantly positively in respect of acid and peroxide values from the hot pressed oils (Table 3). The PV of hot pressed oils (3.05 meq/kg) was higher in comparison to the cold pressed ones (1.98 meq/kg).

The Rancimat test showed that the highest oxidative stability was characteristic for industrial hot pressed oils (Table 3). It results from the fact that a higher temperature of this process on the one hand induces hydrolytic and oxidative processes and on the other hand causes the passing to the oil of such compounds as carotenoids, tocopherols, sterols or phospholipids which are natural antioxidants [Prior *et al.*, 1991a,b]. During the process of seeds conditioning there also appeared the products of non-enzymatic browning which may have the additional anti-oxidant action reducing the negative effect of pro-oxidant compounds. Prior *et al.* [1991b] claim that phospholipids are also responsible for the high stability of pressed oils and that apart from the fact that they alone can provoke anti-oxidant action they may also be treated as potential synergists with other anti-oxidants, *i.e.* with tocopherols. Also phenolic compounds are important for the oxidative stability of pressed rapeseed oils, the content of which decreases in the oils during the refining processes [Koski *et al.*, 2002, 2003].

Heating seeds prior to pressing caused a statistically significant increase in the total content of tocopherols in oils, respectively by about 14% in virgin oils and by nearly 20% in hot pressed industrial oils (Table 3). Despite the change of tocopherol content, the proportions of particular isomers remained the same, there were mostly the  $\gamma$  forms, then  $\alpha$  and only trace amounts of the  $\beta$  forms. The highest content of tocopherols was typical of crude oils. It results from the specificity of the crude oil, which apart from the pressed oil contains also the extracted oil.

### Comparison of cold pressed and fully refined oils

Significant differences appeared in the colour of the tested oils (Table 3). Taking into account the colour determined spectrophotometrically, both carotenoid and chlorophyll groups, the mean values of all the analysed oil types differed in a statistically significant manner ( $p=0.05$ ), (Table 3). Cold pressed oils and virgin oils had an intensive orange hue which was the result of the observed higher content of carotenoid pigments. The ratio between the quantity of carotenoid to chlorophyll pigments was 2.9 in cold pressed oils, 2.2 in virgin type oils and only 0.5 in deodorised oils.

Spectrophotometrical determination of colour proved that the bleaching process caused a decrease in the quantity of pigments by 96% as compared to the crude oils. The deodorization process caused a further removal of pigments. The obtained results of colour evaluation are consistent with those obtained in the literature which show that a properly conducted process of refining causes a colour decrease by about 99% [Płatek & Krygier, 1998]. The removal of carotenoids is disadvantageous from the nutritional point of view whereas chlorophyll pigments are extremely beneficial due to their strong pro-oxidative action in oil.

A low content of chlorophyll expressed as pheophytin a was observed in cold pressed oils compared to virgin, hot pressed and crude oils (Table 3). Pheophytin a was not determined in the deodorised oils because the refining process removes it in almost 100% and the trace quantities are not detectable us-

TABLE 3. Characteristics of the examined oils (6 series).

Characteristics of oils	Cold-pressed	Virgin	Hot-pressed	Crude	Bleached	Deodorized
Spectrophotometric colour 1000( $A_{442} + A_{668}$ )	1085 <sup>a</sup>	1833 <sup>b</sup>	2088 <sup>c</sup>	1914 <sup>d</sup>	78 <sup>e</sup>	18 <sup>f</sup>
$A_{442}$	0.806 <sup>a</sup>	1.253 <sup>b</sup>	1.445 <sup>c</sup>	1.282 <sup>b</sup>	0.065 <sup>d</sup>	0.016 <sup>e</sup>
$A_{668}$	0.279 <sup>a</sup>	0.580 <sup>b</sup>	0.643 <sup>c</sup>	0.632 <sup>c</sup>	0.013 <sup>d</sup>	0.03 <sup>e</sup>
Chlorophyll pigments (mg pheophytin a/kg)	13.92 <sup>a</sup>	45.13 <sup>bc</sup>	45.79 <sup>c</sup>	43.28 <sup>b</sup>	0.25 <sup>d</sup>	-
Acid value (mg KOH/g)	1.92 <sup>a</sup>	2.68 <sup>b</sup>	2.63 <sup>b</sup>	2.70 <sup>b</sup>	0.22 <sup>c</sup>	0.12 <sup>c</sup>
Peroxide value (meq/kg)	1.98 <sup>a</sup>	3.11 <sup>b</sup>	3.05 <sup>b</sup>	3.61 <sup>c</sup>	3.05 <sup>b</sup>	0.79 <sup>d</sup>
Anisidine value	0.61 <sup>a</sup>	0.65 <sup>a</sup>	0.63 <sup>a</sup>	0.79 <sup>a</sup>	2.91 <sup>b</sup>	2.17 <sup>c</sup>
Totox index	4.58 <sup>a</sup>	6.87 <sup>b</sup>	6.74 <sup>b</sup>	8.01 <sup>c</sup>	9.00 <sup>d</sup>	3.74 <sup>e</sup>
Induction time at 120°C in Rancimat test (h)	5.08 <sup>a</sup>	6.57 <sup>b</sup>	7.45 <sup>c</sup>	6.56 <sup>b</sup>	5.33 <sup>ad</sup>	5.37 <sup>d</sup>
Total tocopherols (mg/100 g)	43.1 <sup>a</sup>	49.7 <sup>bc</sup>	51.5 <sup>c</sup>	58.4 <sup>d</sup>	46.3 <sup>ab</sup>	34.3 <sup>e</sup>
$\alpha$ -Tocopherol	14.1 <sup>ab</sup>	17.6 <sup>bc</sup>	18.3 <sup>c</sup>	21.7 <sup>d</sup>	15.9 <sup>bc</sup>	12.8 <sup>a</sup>
$\delta$ -Tocopherol	1.8 <sup>a</sup>	1.5 <sup>bc</sup>	1.6 <sup>bc</sup>	1.5 <sup>bc</sup>	1.4 <sup>bc</sup>	1.1 <sup>d</sup>
$\gamma$ -Tocopherol	27.2 <sup>a</sup>	30.6 <sup>bc</sup>	31.7 <sup>c</sup>	35.2 <sup>d</sup>	29.1 <sup>ab</sup>	20.4 <sup>e</sup>

\* – values marked with the same letter in a row are not significantly different at  $p \leq 0.05$ .

ing the applied method. Virgin type and hot pressed industrial oils contained the largest amounts of pheophytin a. The use of the thermal seed treatment or hot pressing effectively revealed oil colour substances, which was already confirmed by colour evaluation by the spectrophotometric method.

The best quality, because of acid value and peroxide value, is characteristic for the oils after a full refining cycle. At the stage of deodorization free fatty acids were removed as well as the auto-oxidation products, which was reflected in the minimum values of acid and peroxide value. As far as acid value is concerned there is a lack of statistical differences between bleached and deodorised oils; in the case of peroxide value such differences do exist.

Taking into account the anisidine value (the number of secondary products of oxidation) it was noticed that bleached and deodorised oils were characterised by the highest number, and cold pressed and virgin oils by the lowest anisidine value (Table 3). Similar low values for cold pressed and virgin oils were obtained by Górecka *et al.* [2003]. A statistically significant increase of the secondary products of oxidation observed after the bleaching process was confirmed earlier. Also according to the Totox index bleached oils were the worst (the highest values). The deodorization process decreases the value of the peroxide and it is why a decrease of the Totox index was also observed and was the lowest in fully refined oils.

Cold pressed oils showed oxidative stability which differed statistically significant from the stability of the deodorised oils (5.08 h and 5.37 h, respectively) but the difference amounted only 7% on average. However, the initial degree of oxidation of both oils should be taken under consideration, which is significantly higher in the case of the cold pressed oils (Table 3). If the rate of the oxidative changes of both oils were compared using as the starting point the same values of the peroxide value then the dynamics of oxidative changes would proved to be similar and even lower for cold pressed oils. Considering stability differences of the examined oils the following order of the oxidative stability was observed, starting with the most stable: hot pressed oil > virgin type oil > crude oil > deodorized oil > bleached oil > cold pressed oil. In the case of cold pressed oils the untypical course of the oxidative curve was demonstrated without the characteristic period of induction, clearly visible in the case of deodorized oils. It is probably caused by the presence of natural volatile substances in these oils.

The composition of particular isomers of tocopherols was typical of double improved rape seeds [Codex Alimentarius, 2001]. Cold pressed oils were characterised with the average content of tocopherols of 43.1 mg/100 g (Table 3). Deodorised oils contained by about 25% less total tocopherols than the cold pressed oils and by 45% less as compared to the virgin type oils. The total tocopherol losses during the refining process accounted on average for 41%, likewise in other investigations [Kristott, 2000; Gogolewski *et al.*, 2000].

## CONCLUSIONS

1. The method of obtaining oil did not affect the composition of fatty acids and was typical of the low-erucic rapeseed oil. The analysed oils did not contain *trans* isomers of fatty acids with the exception of fully deodorised oils (1.1%).

2. The quality of oils significantly depends on the method of their processing (cold or hot pressing, extraction) and further refining processing. The quality of cold pressed and virgin oils was good and fulfilled the Codex Alimentarius requirements for edible oils. Cold pressed oils differed negatively, in a statistically significant manner, compared to virgin oils and deodorised oils obtained from the same seeds in all examined parameters: colour, acid value, peroxide value, Totox, induction time and only in the case of the anisidine value they showed a beneficially lower value.

3. The highest oxidative stability was observed for hot pressed oils (induction time 50% longer as compared to cold pressed oils). Cold pressed oils were less stable in comparison to oils after full refining.

4. The highest content of tocopherols was observed in crude oils. The refining process caused over 40% decrease of their content in deodorised oils, as compared to their level noted in cold pressed oils. The difference in the nutritive value between cold pressed oils and fully refined oils cannot be stated. However, the 25% higher tocopherol content and the lack of *trans* isomers of fatty acids speak for cold pressed oils.

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Received February 2007. Revision received and accepted June 2007.

## **PORÓWNANIE JAKOŚCI OLEJÓW RZEPAKOWYCH TŁOCZONYCH NA ZIMNO I VIRGIN Z OLEJAMI OTRZYMANymi PRZEMYSŁOWO**

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W pracy oceniono jakość olejów rzepakowych tłoczonych na zimno oraz olejów virgin (z nasion ogrzewanych przed tłoczeniem), uzyskanych w warunkach laboratoryjnych, porównano je z jakością przemysłowych olejów tłoczonych na gorąco, surowych, bielonych oraz odwonionych (po pełnej rafinacji). Metoda otrzymywania oleju nie miała wpływu na skład kwasów tłuszczowych, we wszystkich analizowanych olejach skład ten był typowy dla odmiany rzepaku niskoerukowego. Analizowane oleje nie zawierały izomerów *trans* kwasów tłuszczowych, za wyjątkiem olejów odwonionych (linolowy i linolenowy 1,1%) (tab. 2). Stwierdzono, że jakość analizowanych olejów istotnie zależała od metody ich wydobycia i późniejszego przetwarzania. Jakość olejów tłoczonych na zimno i typu virgin była dobra i spełniały one wymagania stawiane olejom jadalnym. Oleje tłoczone na zimno różniły się statystycznie istotnie od olejów virgin i rafinowanych otrzymanych z tych samych nasion rzepaku, pod względem analizowanych wyróżników: barwy, liczby kwasowej, nadtlencowej, wskaźnika Totox i czasu indukcji w teście Rancimat, z wyjątkiem statystycznie istotnie niższej liczby anizydynowej. Oleje tłoczone na zimno (5,08 h) były mniej stabilne w teście Rancimat w porównaniu do olejów po pełnej rafinacji (5,37 h) (tab. 3). Najwyższą zawartością tokoferoli charakteryzowały się oleje surowe (58,4 mg/100g). Proces rafinacji olejów przyczynił się w efekcie do ponad 40% spadku zawartości tokoferoli w olejach odwanianych. Na korzyść olejów tłoczonych na zimno, w porównaniu do olejów rafinowanych, przemawia o 25% wyższa zawartość tokoferoli i brak izomerów *trans* (tab. 3).