

# Analytic methods to determine the oil content in bran rice in Uruguay

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## INTRODUCTION

The bran rice, is a by-product , obtained during the milled rice production in the industry. It was traditionally used for animal feed in Uruguay, and then, it started to be utilized as raw material, to obtain oil ten years ago. Its price is directly related with its oil content; therefore the importance to obtain a reference analytic method to determine the oil content in bran rice.

The purpose of this poster is to show, how this method is determined, the validation of the extraction using Soxtec Tecator equipment with this reference method and the comparison between the obtained results of both methods using n- hexane and petroleum ether as solvents. The methods that were taken into account as a reference,( AOCS Official Method Ai 3-75 Reapproved 1997 and Revised 1999, AOCS Official Method Aa 4-38 Reapproved 1997 and Revised 2001), were adapted to our objective.

## MATERIALS AND METHODS:

Preparation of sample

A representative sample, is ground to pass a sieve with openings of 1 mm, with a suitable laboratory mill

### Twisselman reference method :

#### Apparatus :

Twisselman extraction apparatus  
Filter paper Whatman 1  
Grinding mill Restch

#### Reagents

n-hexane, gr for analysis

#### Procedure:

A sample of about 5g is accurately weigh and placed into the Twisselman apparatus using 40 ml of n-hexane .  
The extraction time that was determined is 2 hours with 150 or more drops of solvent per minute.  
The solvent is evaporated in an oven at 130 °C to constant weigh.  
( difference between two last mass determinations minor than 5 E -3 g)

### Soxtec method:

#### Apparatus :

Soxtec Tecator apparatus  
Cellulose Whatman extraction thimbles  
catalogue nº 2800266.

#### Reagents :

Petroleum ether, gr for analysis  
n-hexane, gr for analysis

#### Procedure :

A sample of about 5g is accurately weigh and placed into the thimble, in the Soxtec apparatus.  
The extraccion time is : 1 hour in boiling position and 1 hour in rinsing position.  
The solvent is evaporated in an oven at 130 °C to constant weigh.

## OIL CONTENT RESULTS (g/ 100 g) (as is)

Note : all results are without correction by moisture , because there was no difference in the moisture content throughout the time of study, according to the estimated uncertainty of the moisture method determination.

**Table 1-Twisselman reference method  
Extraction time determination**

	t.extrac 1/2 h	t.extrac 1 ht.	t.extrac 1.1/2 hs.	t.extrac 2 hs.	t.extrac 2.1/2 hs.
	17,7890	17,8878	18,4013	18,1724	18,3375
	17,9290	18,0216	18,1480	18,2385	18,3770
	17,7544	18,3200	18,3296	18,4361	18,6635
	17,3550	18,1853	18,1170	18,3808	18,5812
	17,8968	18,2792	18,3334	18,2868	18,4122
	16,7306	18,0814	18,3268	18,5584	18,6016
	17,8250		18,1689	18,5622	18,5693
	17,8340				18,5985
media	17,6392	18,1292	18,2607	18,3765	18,5176
des.std	0,4079	0,1638	0,1125	0,1527	0,1224
varianza	0,1664	0,0268	0,0127	0,0233	0,0150

**Table 2- Comparison between Soxtec method and  
Twisselman reference method**

	T 2h n-Hexane	S 2h n-Hexane
	16,8096	16,7247
	16,8235	16,6632
	16,9562	16,5747
	16,7056	16,7095
	16,9836	16,7824
media	16,8557	16,6909
des.std	0,1142	0,0777
varianza	0,0130	0,0060

**Table 3. Both methods with p. Ether**

	T 2h Petroleum E.	S 2h Petroleum E.
	16,6753	16,5327
	16,6497	16,5687
	16,7196	16,4625
	16,6483	16,6124
		16,5884
		16,5414
media	16,6732	16,5510
des.std	0,0333	0,0524
varianza	0,0011	0,0027

**Table 4- Twisselman method with both solvents**

	T 2h Petroleum E.	T 2h n-Hexane
	16,6753	16,8096
	16,6497	16,8235
	16,7196	16,9562
	16,6483	16,7056
		16,9836
media	16,6732	16,8557
des.std	0,0333	0,1142
varianza	0,0011	0,0130

**Table 5- Soxtec method with both solvents**

	S 2h Petroleum E.	S 2h n-Hexane
	16,5327	16,7247
	16,5687	16,6632
	16,4625	16,5747
	16,6124	16,7095
	16,5884	16,7824
	16,5414	
media	16,5510	16,6909
des.std	0,0524	0,0777
varianza	0,0027	0,0060

## RESULTS DISCUSSION AND CONCLUSIONS

The reference method for the determination of the oil content of bran rice was stated taking into account the Official Method of AOCS and the MGAP decree of Uruguay; this decree recommends an extraction time of 2 hours for sunflower in Butt equipment. Therefore, the extraction times analyzed were the ones in Table 1. The statistic methods used to do the data analysis were variance analysis (ANOVA) ( $\alpha=0,05$ ) and a Student t distribution (two tailed test)( $\alpha=0,05$ ), both of which, showed that, there is no significant difference ( $\alpha=0,05$ ) between an extraction time of an hour and a half and two hours. Even if for the determination of industrial yield is possible to do an extraction of one and a half hours, to accurately determine oil content for reference purpose, is recommended a two hour extraction. The results of oil content obtained with this reference method and the yield historical data of the Uruguayan oil industry are very close.

Table 2 shows the results obtained from the validation of the Soxtec extraction with the reference method. The statistic analysis of data (Student t distribution, two tailed test) showed that in accordance with the estimated uncertainty (0,27g/100g), there is no significative difference between the results obtained from both methods. ( $\alpha=0,05$ ). The same, shows the statistic analysis of data from Table 3.

The statistic analysis (Student t distribution, two tailed test) of data in Table 4 and 5, shows significant difference( $\alpha=0,05$ ) between the results obtained using the reference method or Soxtec extraction with petroleum ether. It is observed that a minor content of oil is extracted when petroleum ether is used.

**REFERENCES:** American Oil Chemists' Society (AOCS) Official Method Ai 3-75. Revised 1999 and Aa 4-38. Revised 2001.

Decreto N°656/986 del Ministerio de Ganadería, Agricultura y Pesca de la República Oriental del Uruguay para girasol.  
Statistic asesor : Sr. José Fuentes.