

Are the commercial automatic devices for oil extraction reliable to be used in ISO 659, the reference standard to determine the oil content in oilseeds ?

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Key norms

International rapeseed commercial transactions are done according to quality parameters such as water, oil and impurities content. For oilseeds and meals, ISO 659 and ISO 22630 are the international standardized oil extraction methods, respectively. In France, NF V03-908 standard where extraction time is reduced (3 h) compared to ISO 659 (8 h) may be used as an alternative for oilseeds. In all these three standards, oil from solid material is extracted with an organic solvent, usually hexane or petroleum ether. Several automatic or semi-automatic devices are now available on the market to facilitate solvent extraction and prevent exposure. To increase operator safety, evaluation of automated system in order to include them in ISO standards used in worldwide trading is thus essential.

Results

Our results show that there is no significant difference neither between values determined on a series of meals (rapeseed or sunflower) nor a series of seeds (rapeseed, linseed, sunflower) with the tested device and the method from ISO 22630 or NF V03-908, respectively. All results show repeatability SD lower than expected by the standards.

Concerning ISO 659, although below repeatability limits defined by the standard, results on Buchi are more variable than noticed in previous analyses.

Even if triple extraction is inducing more manual handling than the two other standards, we notice that Buchi repeatability and intermediate precision SD is always higher to manual ISO 659 regular extraction, especially with the sunflower sample.

Materials and methods

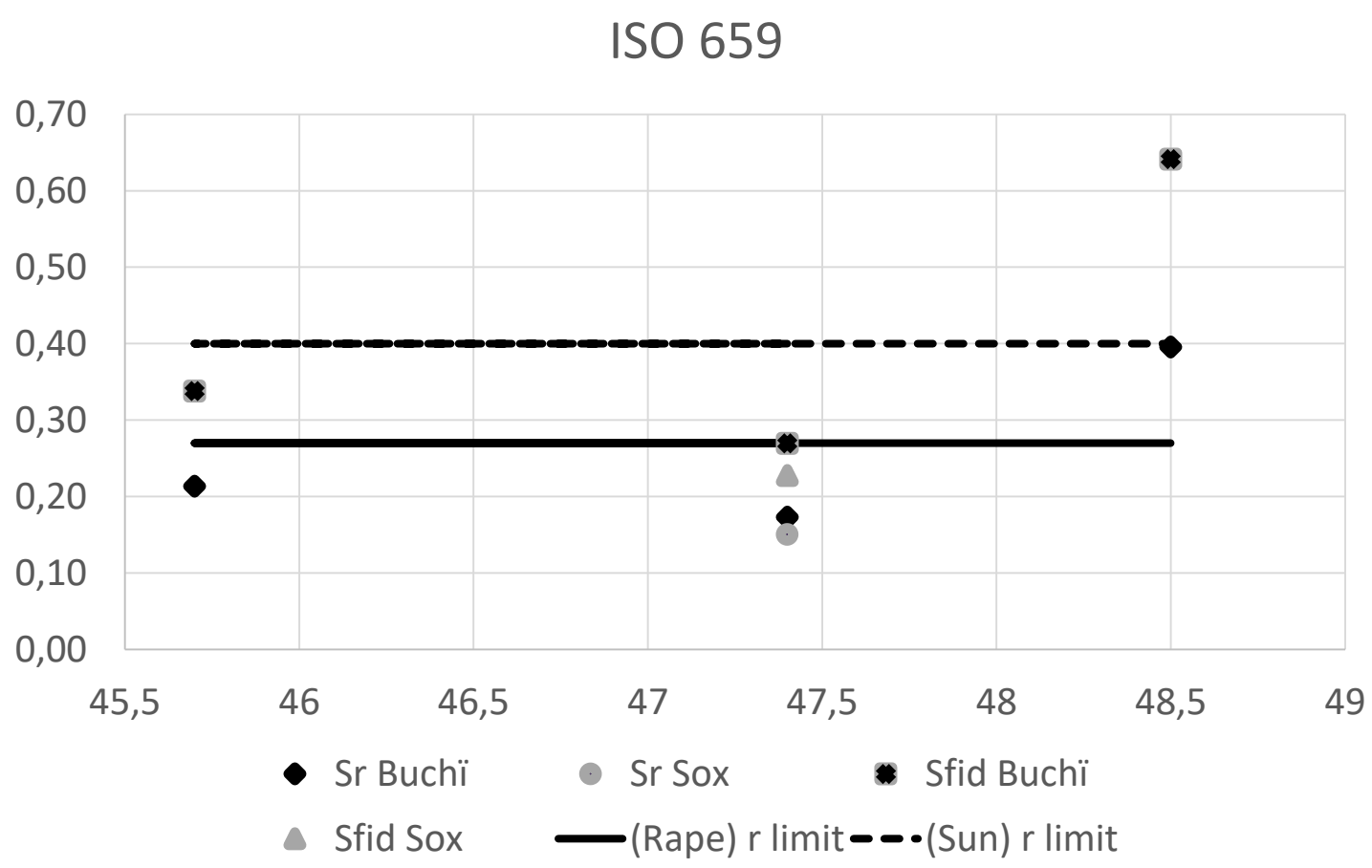
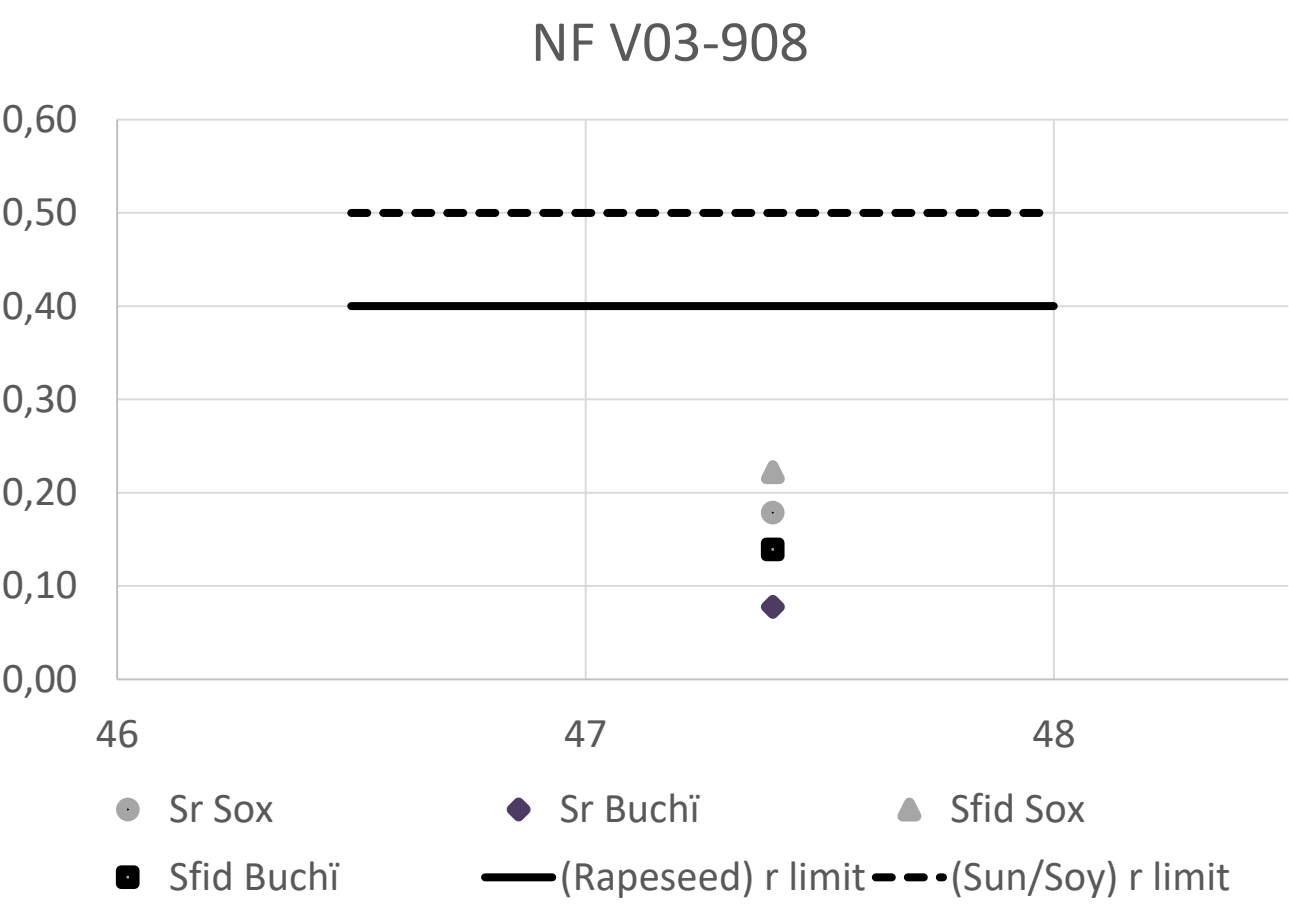
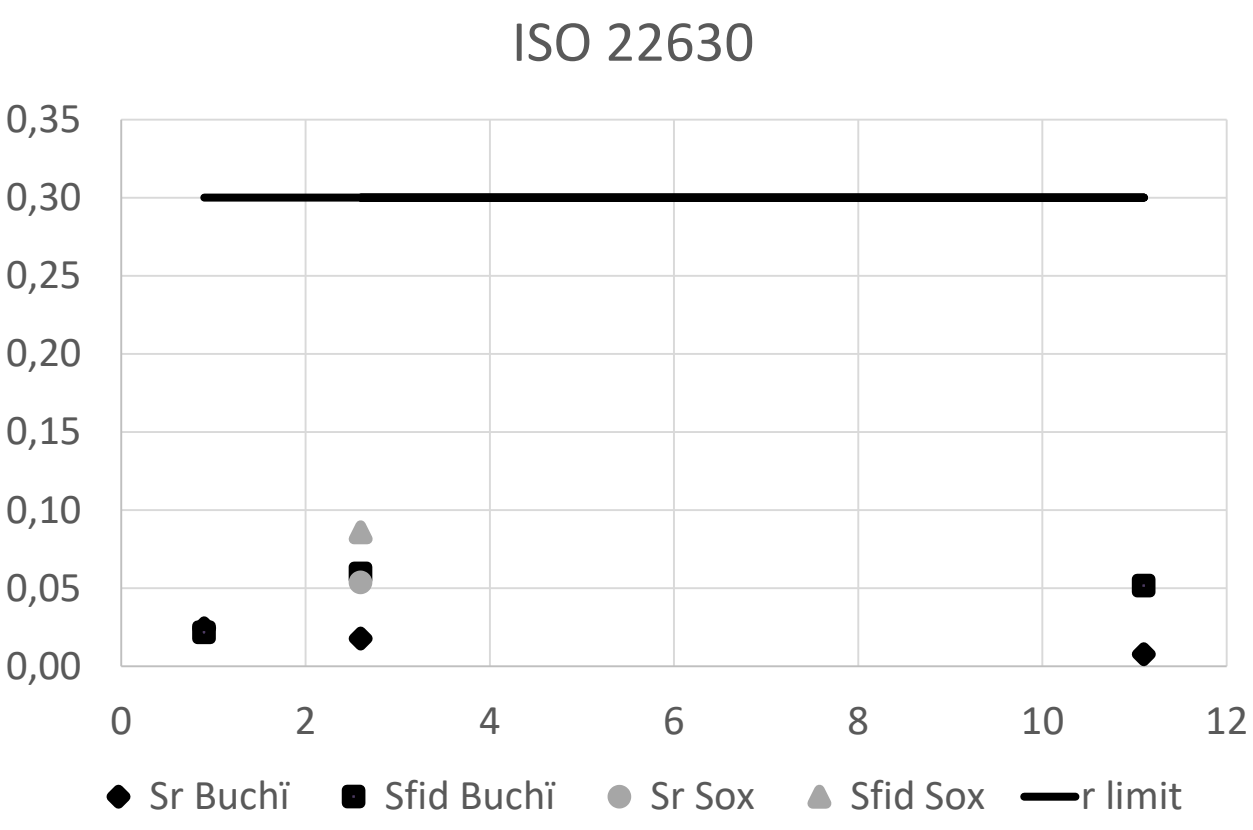
In order to evaluate the device ability to be equivalent to the manual extraction operating mode described in the reference methods (from the three standards cited) and to allow it as an alternative, we have performed a comparative study with one commercial extractor (Büchi B-811).

Several seeds (rapeseed, linseed, sunflower) or meals (rapeseed, sunflower) matrices have been used. All tested samples had defined target values from BIPEA. For each method, at least one sample has been tested according to the regular manual method.

All analyses have been doubled in order to allow repeatability calculation and performed several times by the same operator on different dates in order to allow intermediate precision calculation.

Methods	ISO 22630				NF V03-908		ISO 659			
Manual / Automatic	Manual	Buchi			Manual	Buchi	Manual	Buchi		
Matrix	Rapeseed meals	Rapeseed meals	Rapeseed meals	Sunflower meals	Rapeseed	Rapeseed	Rapeseed	Rapeseed	Linseed	Sunflower
Expected oil content	2,6	2,6	11,1	0,9	47,4	47,4	47,4	47,4	45,7	48,5
Average oil results	2,7	2,7	11,1	1,1	47,1	47,3	47,7	47,8	45,7	48,8
Difference	0,1	0,1	0,0	0,2	-0,3	-0,1	0,3	0,4	0,0	0,3
Repeatability SD, Sr	0,05	0,02	0,01	0,02	0,18	0,08	0,05	0,17	0,21	0,40
Repeatability CV	1,99%	0,65%	0,07%	2,26%	0,38%	0,16%	0,11%	0,36%	0,47%	0,81%
Repeatability, 2,83 Sr	0,15	0,05	0,02	0,07	0,50	0,22	0,15	0,49	0,60	1,12
Nb tests	6	6	3	4	7	6	4	6	3	3
Average oil results	2,7	2,7	11,1	1,1	47,3	47,3	47,5	47,6	45,8	48,8
Difference	0,1	0,1	0,0	0,2	-0,1	-0,1	0,1	0,2	0,1	0,3
Inter. fidelity SD, Sfid	0,09	0,06	0,05	0,02	0,22	0,14	0,19	0,27	0,34	0,64
Inter. fidelity CV	3,19%	2,20%	0,46%	2,01%	0,47%	0,29%	0,41%	0,57%	0,74%	1,31%
Inter. fid., 2,83 Sfid	0,09	0,06	0,01	0,06	0,01	0,01	0,01	0,02	0,02	0,04

1. Table of comparative results according to mentioned standards



2. Relationship between oil content and r SD (Sr) and intermediate fidelity SD (Sfid) by standard or method

Risks and opportunities for ISO 659

In order to investigate this automatic/manual variability, we compared the two systems.

Unlike the regular Soxhlet, Buchi B-811 has a front protection close to extraction chambers, leading to a more confined and thus less ventilated environment. This could keep the solvent warmer in the extraction chamber. To test this hypothesis, we took advantage of a complementary heating system in extraction chamber (two built-in heating sources) available on Buchi B-811 to increase this temperature factor, while reducing extraction time.

Our preliminary results with additional heating (Table 3) show a lower variability than noticed in our previous analyses, but we get slightly higher oil values than expected results. It would be interesting to confirm these results with new analyses (intermediate precision), and with a direct comparison to increased temperature in a regular Soxhlet manual system to validate temperature effect hypothesis. Indeed, if confirmed, this could either be a risk to extract additional compound (to be defined) or to oxidize oil, leading to a false oil content evaluation, or an opportunity to speed up the analysis by reducing the extraction time thanks to a better efficiency.

Methods	ISO 659	Alternatives	
Manual / Automatic	Manual	2h+1h+1h	1h+1h+1h
Matrix	Rapeseed	Rapeseed	Rapeseed
Expected oil content	47,4	47,4	47,4
Average oil results	47,4	48,0	47,8
Difference	0,0	0,6	0,4
Repeatability SD, Sr	0,09	0,1	0,11
Repeatability CV	0,18%	0,16%	0,22%
Repeatability, 2,83 Sr	0,25	0,22	0,30

3. Table of comparative results according to mentioned standards (Manual is 4h+2h+2h)

Conclusions

Our results show that an out of the box automated system provided by Büchi is equivalent to standard extraction on ISO 22630 and NF V03-908. For ISO 659, additional work is needed to understand the differences observed and define the correct method. This work should also be completed by testing other devices to include them in international standards.

We would like to thank Mr EHANNO and Buchi SARL for lending the B-811 to perform these tests.