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PARTICLE SIZE CHARACTERIZATION USING FRAUNHOFFER DIFFRACTION AND MILLING PERFORMANCE OF MAIZE

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Abstract. Twenty-six samples of hybrid maizes (ROSSO flint maize, VOLGA dent maize, DEA and MONARQUE flint-dent maizes) were dry-milled and separated into fractions. The milling yield was determined. Particle size was measured by mechanical sieving for grits, and by laser diffraction for coarse and fine semolina and flours. Bimodal particle size distributions of fine semolina and flours were analyzed as a function of mode size and proportion. Despite inherent variations in culture conditions, differences were apparent between VOLGA and the three other hybrids. The principal mode was characteristic of the fraction considered, without clear varietal differentiation. Analysis of the secondary mode of fine semolina and flours showed that it was principally due to particles similar in size to starch granules (14 to 17 μm), VOLGA particles being smaller than the others. The relative "weight" of the secondary mode was greater with VOLGA than with the three other maize types. The small differences observed between DEA and the ROSSO and MONARQUE hybrids were not significant. A technological behaviour index is proposed: the calculated quantity (g) of very fine particles constituting the secondary modes of fine semolina and flours (per 1 g of ground maize). This index is inversely correlated with the yield of grits (r^2=0.913 for the 26 samples), and may therefore prove useful in the cultivation and processing of maize.

Keywords: maize, varietal selection, milling, fractionation, particle size distribution

INTRODUCTION

The suitability of maize grains for industrial transformations is related to kernel characteristics. Maize endosperm is a storage tissue with a broad variability of physical characteristics in relation to genotype and environmental conditions. These characteristics depend on the amount of horny and floury endosperm. Horny endosperm is formed by small starch granules with a polygonal shape, dipped in a protein matrix of globulin and albumin, and surrounded by protein bodies of zein. Floury endosperm shows larger and round starch granules. Its protein matrix is thinner and no protein bodies are present [2,6,8,18,19].

The two main genetically distinct types of maize are dent and flint. The quantity of horny and floury endosperm and their position on the grain vary according to maize types. Flint maize has a continuous volume of horny endosperm which determines hardness. Flour maize contains nothing but floury endosperm. Dent and flint-dent maize, derived from flint-flour crosses, can differ significantly in their amount of horny and floury endosperm in relation to heredity and environment. Horny endosperm surrounds the floury central part in flint-dent maize. Floury endosperm reaches the kernel crown in dent maize [18]. Dent maize varieties are used in wet-milling and animal feed, flint kernels in dry-milling and cereal flaking.

Dry-milling of maize involves fractionation of particles into variable proportions of grits, flours and middlings. The grains are ground and the fractions are separated according to milling flow diagrams. The coarser endosperm fractions, the hominy grits (3.15 to 6.3 mm) and grits (0.3 to 3 mm), are particularly valuable for the manufacture of breakfast cereal flakes. Finer secondary fractions (semolinas and flours) are used for human foodstuffs after extrusion [1,9]. Middlings and germs are used in the form of fibres and oil, respectively. The mechanical properties of maize depend on the vitreousness of the kernels, and hence on their dent or flint nature [14,20], and on the growth conditions [11,15]. Genetic selection is designed to improve yields of valuable fractions in preparative techniques of milling and separation [4,14].

The milling performance of maize grains is generally characterized by the yield of each separate fraction [10]. More recently, Chaurand *et al.* [5] have developed a pilot mill to determine the suitability of maize for production of hominy grits. In all cases, the fractions are obtained by sieving during milling, and yield depends directly on post-milling particle size. Within the fractions, the size and shape of the particles differ depending on grain hardness [13]. Vitreousness is defined as the ratio of the vitreous to the floury endosperm weight, but considering the difficulties of measurement, the direst determination is never practically made.

The aim of the present study was to compare the particle size of flour and semolina fractions of different types of maize: flint, dent and flint-dent hybrid. Particle size was determined by Fraunhoffer laser diffraction. Compared with mechanical sieving, this measurement technique is very efficient in fine characterization of powders, particularly those with a large range of particle sizes [17]. This additional information may be useful for varietal selection, for adjustment of milling machinery and for nutrition knowledge. Particle size in that case is an indirect method to evalue vitreousness of maize.

MATERIAL AND METHODS

Plant material

Four hybrids were selected:

dent maize was represented by 7 samples of VOLGA,

- flint-dent maize was represented by 7 samples of DEA and 7 samples of MONARQUE,
- flint maize (similar to Plata by reference to the standard Argentine variety) was represented by 5 samples of ROSSO.

The maize types used in this study were grown in variable agronomic conditions and selected through tests set up by INRA.

Test milling

Test milling was performed at the Laboratoire de Technologie des Céréales, INRA (L.T.C.; Cereal Technology Laboratory) under the conditions described by Feillet and Redon [10].

After cleaning, the moisture content of grain (2.5 kg) was brought to 0.16, left unchanged for 24 h, and then increased to 0.19, 30 min before milling.

Milling (Fig. 1) was performed with four grooved rolls for two successive grindings (B1 and B2), followed by degerming (G1) and grinding (B3) of the fraction >2500 μm. Milling yielded five fractions separated by sieving: middlings (>2500 μm), grits (<2500 and >750 μm), coarse semolina (<750 and >400 μm), fine semolina (<400 and >290 μm), and flours (<290 μm). The yield of each fraction was determined. The yield of grits allowed assessment of the dry-milling value of the maize grains (hardness and milling characteristics). Yields were overestimated because the grits were not cleaned and would undergo additional dusting in industrial dry-milling.

Samples of grits, coarse and fine semolina, and flours were kept for studies of particle size.

Particle size measurement

The particles of semolina and flours were sized using a laser diffraction apparatus (Malvern Mastersizer, Malvern Instruments S.A., 30 rue Jean Rostand, Parc Club de l'Université, 91893 Orsay cedex, France) fitted with four focal length lenses, 45, 100, 300 et 1000 mm. The lens focuses the laser light onto a 32-element, concentric, light sensitive ring detector with a hole in the center. Behind the hole is

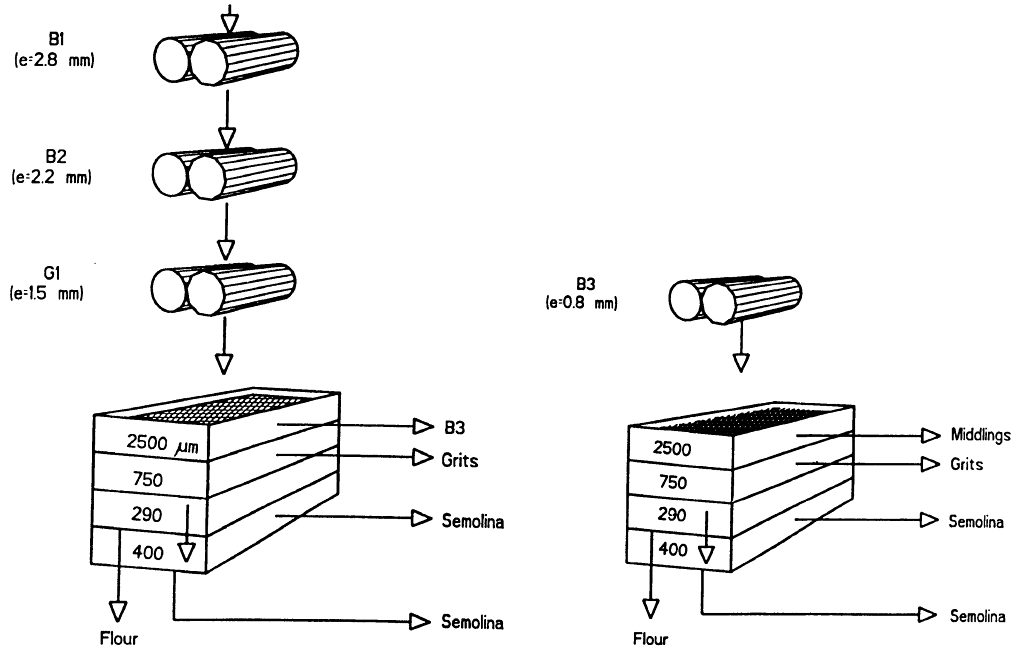

Fig. 1. Flow diagram of test milling: B1, B2, B3 - cylinder mill; e - distance between cylinders; G1 - degerming cylinders. The rectangles represent the sieves with indication of mesh size.

a photodiode used for alignment and for measuring transmittance. The radii of the rings are such that the size ranges covered are 0.1-80 μm, 0.5-170 μm, 1.2-600 μm and 4-2000 μm, respectively. The size range covered by any one lens is divided into 32 logarithmically increasing size classes with the ratio of the largest to smallest diameter about 100:1.

An experiment consist of taking data for background and then for signal plus background. Data are taken by sweeping all 32 rings a number of times. During a single sweep, ring signal are collected serially, digitized and stored in the computer for averaging with the next sweep. The total number of sweeps is user-selectable and is chosen to match the total time the sample will be present in the beam. Typically, a thousand sweeps are taken to ensure that a representative, randomly oriented sample from all size classes has been measured.

At the end of experiment, the average background is substracted from the average signal plus background, and each successive pair of ring data is averaged. These 32 data points are then normalized.

This distribution was related to the measured light intensity, Dj, by the following expression:

$$D_j = \sum_i U_{i,j} V_i$$

where i = the particle size range index, j = the light detection index, Ui,j = matrix describing how the particles in the size range i diffract the light to detector j, Vi = size distribution for the particle range i.

The resulting distribution comprises 32 particle size groups, and their corresponding proportions of volume.

An air-suspended sample (2 x 15 g) was passed through the laser beam, which was diffracted as a function of particle size. The range of measurement was 4 to 2000 μm.

The dry powder feeder is made up of a hopper and a rotating brush. Material feed rate is controlled solely by a vibrator feed rate.

partially dispersed material then enters a cross-flow air injector. The particles encounter a high velocity shear, creating an airborne dispersing action on fine particles. The injector also dilutes the powder aerosol concentration to that required for measurement by diffraction. The powder aerosol then abruptly encounters a 90 degree bend. Large particles centrifuge to the pipe wall and hit it, causing further mechanical dispersion of agglomerates. The large particles are reentrained by the turbulent air flow. The powder aerosol then is ejected across the laser beam through a jet placed in the throat of a vacuum collector. The powder is removed via the vacuum exhaust. The air cell is made up of a ejector nozzle, an optical windows and a vaccuum collector.

Grits size was above the range of measurement of the laser diffraction apparatus and was therefore measured by sieving. A sample (20 g) was placed in a Retsch Vibro 3D apparatus (Bioblock, B.P. 188, 67 405 Illkirch cedex, France) equipped with a series of five sieves of nominal mesh sizes 800, 1000, 1250, 1600 and 2000 μm. At a frequency of 20 Hz, sieving lasted 2 min. The resulting distribution was given in proportion of weight.

RESULTS

Milling yield

Table 1 gives the yields of different fractions produced by milling. The ROSSO, MONARQUE and DEA hybrids gave similar uncorrected yields of grits (>0.70), whereas the VOLGA hybrid gave a lower yield (about 0.64). The flint-dent hybrids (MONARQUE and DEA) and the flint type (ROSSO) gave similar values for this yield. By comparison, the Plata variety, which has reference technological characteristics for the flint type [5], gave a higher yield than MONARQUE (0.76 versus 0.74), whose yield was the highest of the four hybrids studied here. These differences between hybrids are in agreement with observations made by most authors, demonstrating the relation between the vitreousness of the kernel endosperm and the yield of grits [3,13,15,20].

Table 1. Milling yields for the different fractions

Samples	Grits	Coarse semolina	Fine semolina	Flour	Middlings
		VOLGA			
V1	0.668	0.070	0.032	0.031	0.199
V2	0.647	0.069	0.034	0.036	0.214
V3	0.359	0.070	0.032	0.034	0.205
V4	0.629	0.074	0.036	0.039	0.222
V5	0.619	0.072	0.037	0.041	0.231
V6	0.609	0.075	0.038	0.045	0.233
V7	0.641	0.069	0.037	0.037	0.216
		MONARQUE			
M1	0.719	0.063	0.029	0.029	0.160
M2	0.745	0.058	0.026	0.018	0.153
M3	0.741	0.061	0.025	0.020	0.153
M4	0.738	0.059	0.025	0.020	0.158
M5	0.754	0.058	0.024	0.020	0.144
M6	0.747	0.064	0.028	0.022	0.139
M7	0.747	0.057	0.025	0.020	0.151
		DEA			
D1	0.722	0.060	0.027	0.026	0.165
D2	0.723	0.057	0.028	0.028	0.164
D3	0.727	0.056	0.028	0.026	0.163
D4	0.728	0.057	0.028	0.027	0.160
D5	0.743	0.056	0.024	0.025	0.152
D6	0.734	0.058	0.027	0.028	0.153
D7	0.703	0.054	0.027	0.029	0.187
		ROSSO			
R1	0.722	0.060	0.028	0.028	0.162
R2	0.737	0.059	0.024	0.023	0.157
R3	0.742	0.057	0.026	0.022	0.153
R4	0.738	0.056	0.025	0.022	0.159
R5	0.723	0.057	0.029	0.025	0.166

Inversely, the other milling fractions - semolina (0.09-0.11), flour (0.02-0.04) and especially middlings (0.14-0.23) - were produced in greater proportion from the VOLGA type compared with the other hybrids, which were indistinguishable.

Study of particle size distribution

Examples of distribution

An example of particle size distribution for each of the four hybrids is given in Fig. 2 for the grits, coarse semolina, fine semolina and flour, respectively. The distributions are

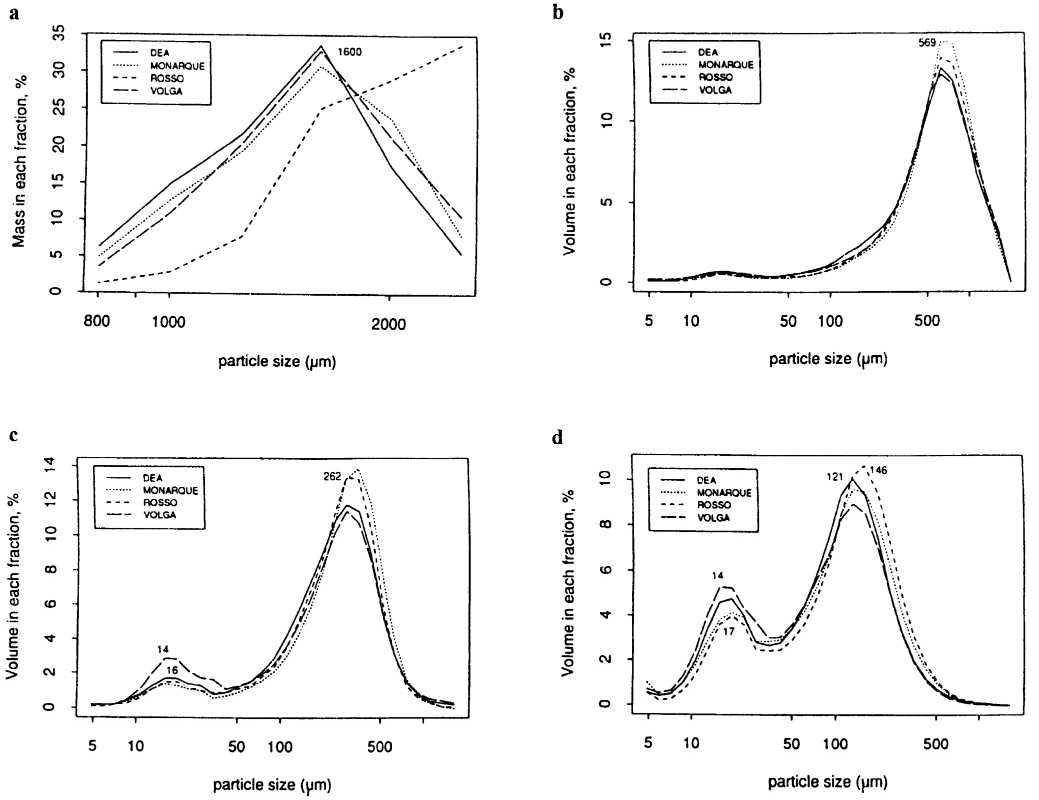

Fig. 2. Particle size distribution of the grits (a), coarse grits (b), fine semolina (c) and flour (d) fractions.

shown using a semi-log scale which provides a more precise description of fine particles. The distributions of grits (Fig. 2a) were similar, except for the ROSSO sample which contained a higher weighed proportion of large particles than the other three samples. The largest fraction was that of 1600 μm overs. The distributions of coarse semolina (Fig. 2b) were also very similar, with a single mode at 569 μm for the four hybrids. The mode of MONARQUE and ROSSO was higher than the one of VOLGA and DEA, for which more particles between 50 and 300 μm were observed. The distributions were bimodal for fine semolina and especially flour, with the principal mode characteristic of the fraction and the secondary mode related to the size of the starch granules. The size of the DEA starch granules was measured by means of image analysis and laser diffraction [7]. The first technique revealed a slightly elongated grain shape (L=21 μm, l=15.2 μm), and the second gave a mean diameter which ranged from 14.5 to 16.5 μm, depending on the cultivating site and conditions. These values for pure starch granules were in agreement with the secondary mode (between 14 and 17 μm) of the fine semolina and flour fractions of DEA. Literature values for starch granule size observed under the microscope are of the same order of magnitude [16].

Each mode of the bimodal distributions of fine semolina and flours was represented by the centre of the most populated class, and the corresponding proportion (Table 2). For fine semolina, the principal mode for the MONARQUE, DEA and ROSSO varieties ranged from 262 to 318 μm, whereas it was more often equal to 262 μm for the VOLGA variety. The respective proportions of the two modes were about 0.90 and 0.10, except for the VOLGA variety (0.84 and 0.16). The principal mode of the MONARQUE, DEA and ROSSO flours ranged from 121 to 146 μm, while that of the VOLGA variety was 121 μm. The respective proportions of the two modes for each variety were about 0.78 and 0.22, except for VOLGA (0.65 and 0.35). One factor variance analyses were achieved to test the effects of the four hybrids on the positions of the mode and on their related ratio (Table 2). Significative differences were observed for all parameters with the exception of the position of mode 2 in the case of fine semolina. The proportions associated to modes 1 were found to highly depend on the hybrids both for flour and fine semolina. The corresponding multiple range analyses showed that this effect was caused by VOLGA and no significative differences were observed for the three other hybrids (Table 3). Similar results were found concerning the position of mode 2 in the flour case. The position of mode 1 depended in a less extent on the hybrids both for fine semolina and flour. However, no separated homogeneous groups of hybrids were revealed. VOLGA was therefore distinct from the other varieties mainly regarding the proportions of modes 1. For fine semolina, the proportion of the secondary mode was higher for Volga than for the 3 other hybrids. This tendency was confirmed and increased with the flours. The secondary mode related to the starch granules reached 0.40 of the total flour volume with VOLGA, whereas it was about 0.20 for ROSSO. These results show that a milling fraction may exhibit a variable particle size distribution, revealed here by laser diffraction measurements.

DISCUSSION

Quantitative differences in milling yields were essentially related to whether the maize was a flint or dent type. Our aims were to analyze particle size and to determine whether there were qualitative differences. Conventional analysis of particle size distribution by mechanical sieving would not have revealed a significant difference between VOLGA and

Table 2. Values and proportions of the modes in the bimodal distributions for fine semolina and flour

Samples	Flour			Fine semolina		
	mode					
	1		2	1		2
	(μm)	ratio (1)	(μm)	(μm)	ratio (1)	(μm)
			VOLGA		*	
V1	121	0.71	17.3	262	0.89	15.6
V2	121	0.62	14.2	262	0.81	14.2
V3	115	0.63	14.2	262	0.80	14.2
V4	121	0.60	14.2	262	0.84	17.3
V5	121	0.71	16.4	262	0.86	14.9
V6	121	0.64	14.2	262	0.85	14.2
V7	121	0.67	14.2	274	0.84	14.2
			MONARQUE			
M1	121	0.74	17.3	274	0.92	16.4
M2	146	0.81	17.3	318	0.92	17.3
M3	126	0.78	17.3	318	0.93	14.9
M4	139	0.78	17.3	318	0.93	14.9
M5	146	0.81	17.3	318	0.93	15.6
M6	126	0.76	17.3	318	0.90	14.9
M7	139	0.78	17.3	318	0.89	17.3
			DEA			
D1	132	0.74	17.3	262	0.91	17.3
D2	121	0.73	17.3	262	0.90	17.3
D3	121	0.76	17.3	262	0.91	15.6
D4	121	0.77	17.3	318	0.92	14.3
D5	121	0.76	17.3	262	0.91	17.3
D6	139	0.78	17.3	274	0.88	14.9
D7	126	0.77	17.3	262	0.89	17.3
			ROSSO			
R1	121	0.76	17.3	262	0.91	18.1
R2	146	0.77	17.3	262	0.91	17.3
R3	146	0.82	17.3	287	0.93	15.6
R4	126	0.78	17.3	318	0.93	15.6
R5	146	0.81	17.3	318	0.93	15.6
F-ratio	5.4	28.4	19.7	9.2	23.4	2.1
Sig. level (2)	*	***	***	**	***	NS

(1) proportion assosiated with the mode, (2) analysis of variance, effect of variety, significance level (%), 0.1***; 1**; 5*.

Table 3. Ratio mode 1. Multiple range analysis using the Least Significative Difference (LSD) with a significance level of 95%. Crosses indicate homogeneous groups

	Flour	Fine semolina
VOLGA	0.65	0.84
DEA	0.76 X	0.90 X
MONARQUE	0.78 X	0.92 X
ROSSO	0.79 X	0.92 X

the other hybrids, because their principal modes were often equivalent (Table 2). Laser diffraction analysis, on the other hand, gave a good characterization of the fine fraction, revealing a secondary mode of variable proportion for the fine semolina and flours. This secondary mode was most probably due to individual starch granules dissociated from endosperm proteins during milling. Likewise, the usual expression of the results as a mean diameter and a deviation would not conveniently represent the particle size distribution, whereas spectral characteristics (modes) are more suitable. It was the relative "weight" of the secondary mode (cf. principal mode) which was important, and not its intrinsic size. Otherwise FCA (Factorial Correspondence Analysis) made it possible to represent all 26 samples on a single map, and to take all size distributions into account. The maps revealed differences between VOLGA and the other hybrids for the finest fractions [12].

The results (Table 2) indicated that the proportion of particles considered as starch granules depended on the flint or dent nature of the hybrids. This information combined with the milling yields (Table 1) could be used to characterize the samples more completely. Quantification of the secondary modes may allow calculation of a ratio which could be a "technological behaviour index" for maize. Indeed, the combined proportions of the secondary modes for fine semolina and flour (for 1 g of ground maize) were strongly correlated (r^2 = 0.913) with the yield of grits (Fig. 3). The proportion of fine particles considered as individual starch granules was lower at high yields. As before, VOLGA was clearly separated from DEA, ROSSO and MONARQUE, which were grouped together. This index could be proposed to characterize the influence of the maize variety and growth conditions. Grain hardness and millability could also be linked to this index. It would be interesting to see if the observed particle size differences in a given milled fraction are related to behaviour, such as liquid uptake capacity and suitability for shaping, during secondary processing. Otherwise genotype of maize could influence upon rate of ruminal starch degradation related to variable proportions of fines particles of the flour.

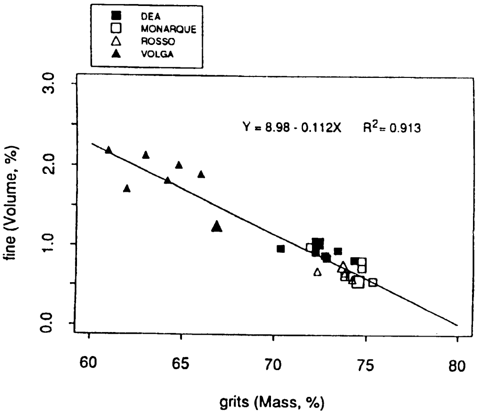

Fig. 3. Relation between the quality of fine particles considered as starch granules and the yield of grits from ground maize.

CONCLUSIONS

Particle size analysis by laser diffraction of the milled fractions of four types of maize (flint/dent) revealed a bimodal distribution of the finer fractions (semolina and flour), whose secondary mode was related to the size of separate starch granules. The proportion of this secondary mode was higher for VOLGA dent maize and varied inversely with the yield of grits for all samples. A clear difference between the VOLGA dent maize and the other hybrids (flint and flint-dent types) could be observed. These modern measurement improve the characterization of particle size distribution. They also represent an alternative behavioural measure of the technological suitability of a milled product (pooled or individual fractions).

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