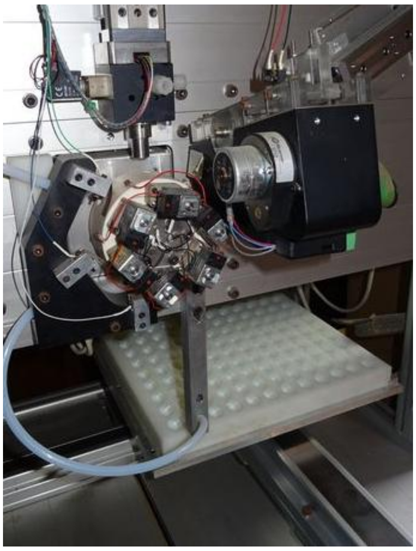
SELECTION OF CAPILLARIES FOR THE PRODUCTION OF "EQUAL CAPILLARIES TYPE" DILUTERS



In the manufacturing process of glass capillaries the internal diameter (capillary hole) necessarily has a certain tolerance and therefore presents a certain dispersion, which is normally specified in the purchase order.
When a batch of capillaries is used as a "calibrated flow resistance", the induced flows may be calculated depending on the fourth power of the diameter and the dispersion of the values in the lot increases considerably.
It is certainly necessary that the selection of the capillaries destined to be used in an "equal capillary" diluter is preceded by a classification operation, in which the capillaries with similar characteristics in a certain tolerance band are grouped into separate classes.
The classification test is performed on each capillary by measuring the flow induced by a stable pressure applied to one end of the capillary.
Initially the test was manual, but regardless of the time required to classify a thousand or more capillaries for each purchase lot, it was subject to important errors due to the heat transferred from the operator's hands to the tool and to the capillary during the insertion in the measuring circuit (the viscosity depends on the temperature of the gas).

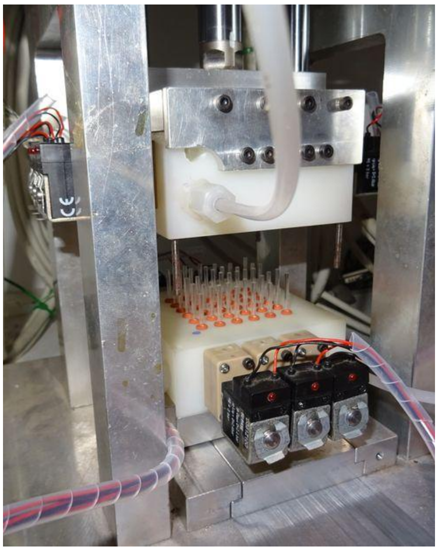
Today the manual practice is surely abandoned, but we are still using and trying to improve different tools. This activity is necessary not so frequently as to force us to tackle the problem decisively, but the fact remains that all purchased capillaries are classified and then sorted in "flow resistance" classes in ascending order, and each class contains a range of values with a high probability of being contained in a narrow band.

<u>Final selection, applied to build BetaCAP30</u>

The resulting precision after selection is generally not sufficient to meet our needs (we want to use all the potentials inherent in using "equal capillaries"). We have therefore created a second tool for the final selection in which the number of capillaries (and therefore the selection times) are more contained. Each selection is made by processing one or a few pre-selected classes to give rise to a single diluter. The instrument used for this purpose can test 36 capillaries placed in a 6x6 matrix with 6 inlet solenoid valves (rows) and 6 discharge solenoid valves (columns) organized therefore in a way that allows both the measurement of capillary sub-matrices (used just for the pre-scaler BetaCAP1A100) and of one capillary at a time. It should be noted that, while the six inputs (rows) are intercepted by two-way solenoid valves (open / closed), the output solenoid valves (columns) are three-way type (connection to the meter / venting in air): the way that connects the column at the vent is necessary to depressurize the 5 columns not destinated to the measurement. In the absence of this depressurization parasitic flows would be created crossing all capillaries on the active row and closing in the active column crossing capillaries other than the one "in measure", which is located at the intersection of the active row and the active column. The flow that crosses the capillaries not "in measure" of the selected row, is instead drained to the outside, keeping all the columns at ambient pressure.

The matrix is then scanned to determine 36 values, one for each capillary: the homogeneity of the selected batch is verified and, in the case of values out of tolerance, the corresponding capillaries are replaced. If necessary the operation is repeated until the best homogeneity of the lot is reached.

It is useful to add now some elements to the modalities of execution of the "measures" indicated between the quotes just because they are not definable measures to all effects: having the purpose of determining "how much two or more flows evaluations are different relative to themselves", it is



sufficient to get from the measuring system just numbers (even without considering the measuring units) which, however, we know to be proportional to the corresponding flow values.

The data that we must treat with the utmost attention is the repeatability of the relief, which includes the repeatability of the pressure applied to the capillary. The knowledge of the Bias, understood as a systematic relationship between the number detected and the magnitude applied to the measurement system, adds nothing to what is necessary to quantify the relative differences between the flows of the different capillaries. The answer of our measurement system therefore remains, from a metrological point of view, absolutely unknown.

A great deal of attention is instead devoted to the verifications and corrections of everything that acts on repeatability: drift of the meter and of the applied pressure value. The capillary in position R1C1 (first row, first column) is given the qualification of "internal reference": the applied pressure value (unknown) is increased until the number representing the flow reaches a value of the desired consistency. This is in the order of 300.0 ... 400.0, which allows us to appreciate the first decimal with a typical oscillation of ± 1 which corresponds to less than one per thousand of the number itself.

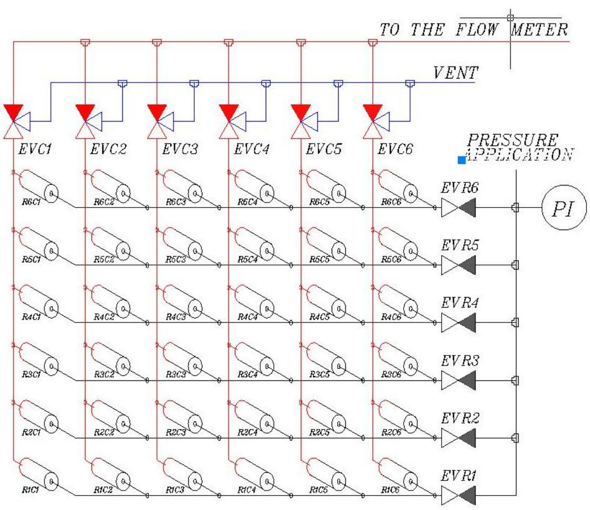
The matrix is scanned by sequentially detecting the "flows" on the 6 capillaries (six rows) of a column, to then continue with the 6 capillaries of the second column, and so on up to the sixth column, sixth row.

The "flow" of the reference capillary is checked every 6 "measurements", together with the value of the applied pressure. This choice is motivated by two facts:

a) moving along the column, the flow supplied by the inlet regulator is constant: what changes is only the proportion between the flow directed to the meter and the flow directed to vent. Providing a constant flow the pressure regulator is definitely stable and, in the time of 6 "measurements" (the 6 lines) the drift of the meter is negligible (having initially expected a stabilization time for the whole system).

b) passing to the next column, 6 different capillaries are fed which, although homogeneous, can absorb a slightly different flow: this difference is sufficient to influence the applied pressure. Moreover, in the scanning time of more columns it is possible to begin to manifest a drift of the measurement system that should be corrected.

At the end of the scanning of each line, the reference capillary is then selected in pos. R1C1 verifying if the "measurement" corresponds to the initial value: if necessary the value of the applied pressure is modified to restore the initial "measuring" value. It may surprise you that you act on the pressure when it is not known if the drift is due to the meter or to the pressure regulation: in fact it is sufficient that the "measurement" of the reference capillary returns to the initial conditions, so that all the others can continue to be effectively compared with it. The minimum variation in sensitivity of the meter (assuming it is detectable) has negligible effects and only on the extent of the deviations detected (we are talking about decimals% of decimals% = few ppm).



Moving on to the next column, the applied pressure can have an immediate change: it must be restored to the pressure value noted at the end of the previous "calibration" with the reference capillary.

Each of the 36 capillaries of the test matrix is therefore characterized by a value proportional to the flow detected with the same applied pressure. The test is repeated three times (Columns A, B, C) and of the three values corresponding to each capillary the two closest to each other are averaged.

The more "distant" value is discarded because the anomaly could also be due to a transcription error, which inserted on average could have false and not negligible effects.

The average values of the "measured" flow, from column F are reported in column J and the corresponding coordinates from column A to column I. The two columns I and F are ordered by increasing flows.

A "measured" flow value is identified close to the average value of the flows and in column K the difference between the corresponding measured value and the identified value is indicated.

Column K will therefore contain the deviations of each "measure" with respect to the average value of the "measures": a good part will be negative and another will be positive.

Now the selection of the capillaries to compose the groups (from 1, 2, 4, 8, 15 capillaries) that make up the BetaCAP30 diluter :

a) the group with 1 capillary will contain one of the capillaries which in column K corresponds to a zero ("Measured" flow value nearly equal to the average value)

b) the group with 2 capillaries will contain two capillaries which in column K correspond to equal numbers but of opposite sign: one is scarce and the other abundant for the same quantity with respect to the single capillary selected. What matters is that, with the same applied pressure, the flow induced in the group by two capillaries is double that the one induced in the single capillary.

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q
1																	
2		Be.T.A.															
3		Strumentazione Srl							Just gas dividers, but								
4																	
5		CAPILLARIES SELECTION CHART													S/N		22/08/2018
6																	
7	Position.	Meas.A	Meas.B	Meas.C	too far	Average	Max		ordered		Dev. From	Destin.					
8					exclud.	of near	Deviation		position		Average	group					
9															Q.ty to	totale	Selected
10	11	358,60	358,60	358,60	358,60	358,60	0,000		13	349,30	-5,70	15			be sel.	deviation	q.ty
11	12	350,90	351,60	351,10	351,60	351,00	0,057		12	351,00	-4,00	15		Group of	1	0,00000	1
12	13	349,20	349,30	349,30	349,20	349,30	0,000		34	351,50	-3,50	8		Group of	2	0,00000	2
13	14	351,70	351,90	352,00	351,70	352,00	0,028		21	351,60	-3,40	15		Group of	4	0,00000	4
14	15	351,40	351,90	351,80	351,40	351,90	0,028		15	351,90	-3,10	15		Group of	8	0,00000	8
15	16	351,90	352,10	352,50	352,50	352,00	0,057		14	352,00	-3,00	15		Group of	15	0,00000	15
16	21	352,00	351,50	351,60	352,00	351,60	0,028		16	352,00	-3,00	15					
17	22	351,60	351,90	352,00	351,60	352,00	0,028		22	352,00	-3,00	15					
18	23	354,10	354,20	354,40	354,40	354,20	0,028		23	354,20	-0,80	4					
19	24	354,10	354,30	354,40	354,10	354,40	0,028		25	354,20	-0,80	4					
20	25	353,80	354,10	354,20	353,80	354,20	0,028		31	354,30	-0,70	8					
21	26	354,60	354,90	355,10	354,60	355,00	0,056		33	354,30	-0,70	8					
22	31	354,70	354,30	354,30	354,70	354,30	0,000		24	354,40	-0,60	2					
23	32	355,10	355,50	355,40	355,10	355,50	0,028		26	355,00	0,00	1					
24	33	354,20	354,30	354,50	354,50	354,30	0,028		35	355,30	0,30	8					
25	34	351,40	351,50	351,70	351,70	351,50	0,028		32	355,50	0,50	8					
26	35	354,80	355,20	355,40	354,80	355,30	0,056		41	355,50	0,50	4					
27	36	355,50	355,70	356,10	356,10	355,60	0,056		36	355,60	0,60	2					
28	41	355,40	355,50	355,60	355,60	355,50	0,028		45	356,10	1,10	4					
29	42	355,60	356,30	356,10	355,60	356,20	0,056		42	356,20	1,20	8					
30	43	357,20	357,40	357,60	357,60	357,30	0,056		51	356,40	1,40	8					
31	44	356,50	356,90	357,00	356,50	357,00	0,028		53	356,50	1,50	8					
32	45	355,70	356,10	356,10	355,70	356,10	0,000		56	356,90	1,90	15					
33	46	356,60	356,90	357,20	356,60	357,10	0,084		61	356,90	1,90						
34	51	356,30	356,10	356,40	356,10	356,40	0,028		44	357,00	2,00						
35	52	356,60	357,00	357,10	356,60	357,10	0,028		46	357,10	2,10						
36	53	356,40	356,60	356,80	356,80	356,50	0,056		52	357,10	2,10						
37	54	357,20	357,40	357,60	357,60	357,30	0,056		43	357,30	2,30						
38	55	357,40	357,80	357,80	357,40	357,80	0,000		54	357,30	2,30	15					
39	56	356,50	356,80	357,00	356,50	356,90	0,056		63	357,70	2,70						
40	61	356,90	356,90	357,10	357,10	356,90	0,000		55	357,80	2,80	15					
41	62	357,30	358,00	357,90	357,30	358,00	0,028		62	358,00	3,00	15					
42	63	357,60	357,70	357,80	357,80	357,70	0,028		64	358,30	3,30	15					
43	64	358,00	358,20	358,30	358,00	358,30	0,028		11	358,60	3,60	15					
44	65	359,20	359,40	359,50	359,20	359,50	0,028		66	358,80	3,80	15					
45	66	358,60	358,90	359,30	359,30	358,80	0,084		65	359,50	4,50	15					
46																	
47						Max dev%	0,084		Media :	355,3639							

c, d ...) groups of higher order, will be composed in the same way, generally choosing half of the needed capillaries scarce and the other half abundant so that the sum of the deviations is null.

The choices are formalized by writing at col. L the number of capillaries making up the group at each selected capillary

The columns P and Q, analyzing the contents of columns K and L assist the operator by checking that the quantities selected correspond to what is necessary both in terms of numerical consistency and of deviation balance.

The most important point of this procedure concerns the selection of the capillaries to be inserted in the smaller groups (1, 2, maximum 4 capillaries), while for groups containing 8 or 15 capillaries the accuracy of the selection is however exaggerated: we are managing deviations in the order of 1 per thousand for the single capillary, which are combined with the laws of statistics for the largest groups, but in relative terms must be reported at 8 or 15 times greater flows!

Knowing the deviation values between the nearest measurements (repeatability index) it is preferable not to use in the specific selection (6 capillaries still advance) the capillaries corresponding to the higher deviations, or at worst use them to compose the larger groups.

At the end of construction, each product produced, if requested by the Customer, is subjected to the metrological test: Accredited companies for flow and pressure measurements, in the measurement range necessary for the purpose, perform the necessary traceable measures to make the product legally usable in accredited environments (LAT).

In the case of the BetaCAP30 diluter, 7 tests are performed in which, by setting dilutions 0:30, 1:30, 2:30, 4:30, 8:30, 15:30, 30:30, the incoming flow is measured to the TG1 connection (gas to be diluted) and the outgoing flow (diluted output flow). The ratio between the two measurements corresponds to the dilution ratio and the deviations are calculated as the difference between the dilution measured as a flows ratio and the dilution calculated as the numerical capillaries ratio Number of span gas crossed capillaries / Total installed capillaries (N/30).

In the absence of significant leakages (TG1 = 0 for dilution 0:30 and TG0 = 0 for dilution 30:30) the flows measured in correspondence with the 5 main ratios (those that see only one group of capillaries at a time affected by TG1) characterize the systematic response of the diluter in all his modes of operation (31 dilution ratios between 0 and 100%), which correspond to the different combinations of the 5 main ratios.