preferably mixed and ground in solid form. They are subsequently dissolved by addition of water or an aqueous buffer. Whereas hygroscopic or unstable components may have to be stored and brought into solution separately, the compacted glycine granules can readily be processed with the other stable components.

The present invention also relates to a process for increasing the storage stability of glycine powder, in particular for retaining the flowability of glycine. In this process, glycine powder is compacted to give glycine granules, where at least 75% (w/w) of the glycine granules obtained have a particle size of at least 0.7 mm.
The preferred embodiments of the preparation process of compacted glycine granules mentioned above apply here. The process can be carried out at room temperature. The glycine granules obtained by means of the process have a significantly higher storage stability than the glycine powder employed as starting material. In particular, the flowability is maintained for longer compared with glycine powder under identical storage conditions.

The processes according to the invention are simple and effective to carry out. Without a chemical change to the glycine, the properties of the glycine can be modified purely by mechanical actions, such as pressing and optionally sieving. In particular, a significant increase in the storage stability takes place, since the material according to the invention cakes significantly more slowly and remains flowable for longer.

Even without further comments, it is assumed that a person skilled in the art will be able to utilise the above description in the broadest scope. The preferred embodiments and examples should therefore merely be regarded as descriptive disclosure which is absolutely not limiting in any way.

Examples

A) Equipment and methods for characterisation of the substance properties

1. Bulk density: in accordance with DIN EN ISO 60: 1999 (German version)
   -figures in "g/ml"

2. Tapped density: in accordance with DIN EN ISO 787-11: 1995 (German version)
   -figures in "g/ml"

3. Compressibility index: in accordance with 2.9.36. Powder Flow Ph Eur 8.0 (English version)
   -figures in "%"

4. Hausner factor: in accordance with 2.9.36. Powder Flow Ph Eur 8.0 (English version)
   -dimensionless

5. Particle size determination by dry sieving through a sieve tower: Retsch AS 200 control, Retsch (Germany); amount of substance: about 110.00 g; sieving time: 30 minutes; amplitude intensity: 1mm; interval: 5 seconds; analytical sieves with metal wire mesh in accordance with DIN ISO 3310
   -sieve widths (in µm) for comparison 1 and 2: 1000, 710, 600, 500, 400, 355, 300, 250, 200, 150, 100, 50, 32
   -sieve widths (in µm) for Example A, B, C, D, E and F: 2000, 1700, 1600, 1400, 1250, 1120, 1000, 900, 800, 710, 600, 500
   -Indication of the amount distribution per sieve fraction in the tables as "% by weight of the sample weight"

6.Determination of the drying loss:
Determination according to Ph Eur 8.6 in accordance with glycine monograph: dry 1.000 g of substance at 105°C for 2 hours in the oven (Ph Eur 8.6 under 2.2.32.)
-figures in "% by weight"

7.DVS conditions (determination of the hygroscopicity):
Surface Measurement Systems Ltd. UK 1996 – 2007, Method: 0-98% RH, 10% steps, 25°C, 0.0005wt%-min., halfcycle.sao; performance of the measurement in accordance with manufacturer's instructions
-increase in weight figures in "% by weight"

8. Dissolution rate:
Equipment used: Mettler AT201 balance, 150 ml beaker, IKA® RCT basic magnetic stirrer plate, 50 ml measuring cylinder, stirrer bar diameter 7 mm and length 4 cm, stirring speed 200 rpm, amount of substance 4.00g+/-0.1g, DI water with a temperature between 20 and 25°C ("DI water" is "deionised water");
Procedure: place 50ml of DI water in the beaker and switch on the stirrer. Place the thermometer in the DI water, add the substance and measure the time (stopwatch) until the substance has visually dissolved without a residue.
-figures in "seconds"

9.Storage conditions in climatic cabinets:

a) Comparisons 1 and 2 and Examples E and F: in each case 160g+/-5g of substance are stored at 25°C/60%RH and 40°C/75% RH both open (in a glass dish) and also closed (in a screw-lid glass container) – the caking is assessed after a storage time of 1, 2 and 7 weeks.
- Glass dish: diameter 95mm, height 55mm, the substance is distributed over the dish base in a uniform layer thickness; the assessment is carried

out by tilting the dish and visually observing the flow behaviour depending on the tilt angle (in some cases, however, the samples have already caked so much that free powder flow can no longer be observed)
- screw-lid glass container: 250ml, height 11.5cm, outside diameter 7cm, white glass tightly sealed with plastic screw lid, the assessment is carried out by tilting the glass and visually observing the flow behaviour depending on the tilt angle (in some cases, however, the samples have already caked so much that free powder flow can no longer be observed – in these cases, after rotation of the glass through 180°, the glycine remains adhering to the glass base or only deposits in part)

b) Examples A, B, C and D: in each case 120g+/-5g of substance are stored at 40°C/75% RH both open (in a glass dish) and also closed (in a screw-lid glass container) – the caking is assessed after a storage time of 2 and 7 weeks.
- Glass dish: diameter 95mm, height 55mm, the substance is distributed over the dish base in a uniform layer thickness; the assessment is carried out by tilting the dish and visually observing the flow behaviour depending on the tilt angle (in some cases, however, the samples have already caked so much that free powder flow can no longer be observed)
- screw-lid glass container: 250ml, height 11.5cm, outside diameter 7cm, white glass tightly sealed with plastic screw lid, the assessment is carried out by tilting the glass and visually observing the flow behaviour depending on the tilt angle (in some cases, however, the samples have already caked so much that free powder flow can no longer be observed – in these cases, after rotation of the glass through 180°, the glycine remains adhering to the glass base or only deposits in part)

10.Weight changes after storage
-weight change figures in “g” compared with the initial value at the beginning of storage

11."Break energy" determination:
REVOLUTION powder analyzer (Mercury Scientific Inc, Newton, USA); rotation rate 0.3rpm, test method FlowMethod_SP.fam; drum diameter 100 mm; amount of powder 95-100 ml
-figures in "mJ"

12. "Avalanche angle" determination:
REVOLUTION powder analyzer (Mercury Scientific Inc, Newton, USA); rotation rate 0.3rpm, test method FlowMethod_SP.fam; drum diameter 100 mm; amount of powder 95-100 ml
-figures in degrees ("°")

B) Working methods

Commercially available crystalline pulverulent glycine (Article 100590 glycine cryst. suitable for use as excipients EMPROVE® exp Ph Eur, BP, JP, USP from Merck KGaA, Darmstadt, Germany) is subjected to dry granulation over roller compactors with subsequent crushing and sieving via an oscillating sieve mill.
Two different compactors are used:

1.Examples A to D: model RC 100 compactor from powtec Maschinen and Engineering, Remscheid, Germany; roller diameter 100 mm, roller width 30 mm, grooved roller surface, no roller cooling, sieve mesh width 3 mm, undersized particle sieving 1 mm without material recycling; the gap width arises through the roller pressure selected

2.Examples E and F: model K200/100 compactor with FC 400 sieve mill from Hosokawa Bepex, Leingarten, Germany; roller diameter 200 mm, roller width 100 mm, grooved roller surface, no roller cooling, sieve mesh width 2 mm; undersized particle sieving 1 mm with material recycling; the gap width arises through the roller pressure selected

The comparison against the prior art takes place against 2 batches of a commercially available crystalline pulverulent glycine from Merck KGaA, Darmstadt (Germany), article 100590 glycine cryst..

1.Examples A, B, C and D: preparation of a compacted glycine having improved flowability and storage stability (compacted glycine having coarse fractions >2000 µm prepared under various roller pressures)

Compaction Example A:

speed of rotation of screw 30 rpm, speed of rotation of rollers 5 rpm, pressure at the pressure rollers 5.23 +/- 0.33 KN/cm

Compaction Example B:

speed of rotation of screw 30 rpm, speed of rotation of rollers 5 rpm, pressure at the pressure rollers 6.54 +/- 0.33 KN/cm

Compaction Example C:

speed of rotation of screw 20 rpm, speed of rotation of rollers 4 rpm, pressure at the pressure rollers 7.84 +/- 0.33 KN/cm

Compaction Example D:

speed of rotation of screw 30 rpm, speed of rotation of rollers 5 rpm, pressure at the pressure rollers 3.92 +/- 0.33 KN/cm

rpm = revolutions per minute

Bulk density, tapped density, Carr index:

(details on the measurement methods, see under Methods)

Sample	Bulk density (g/ml)	Tapped density (g/ml)	Hausner factor	Compressibility index (%)	Drying loss (%)
Example A	0.72	0.82	1.14	12.7	0.02
Example B	0.72	0.82	1.13	11.4	0.04
Example C	0.72	0.80	1.12	10.5	0.05
Example D	0.73	0.82	1.12	10.6	0.04

Particle distribution determined by tower sieving:

Figures in % by weight (details on the measurement method, see under Methods)

Sample	<500 µm	500-600 µm	600-710 µm	710-800 µm	800-900 µm	900-1000 µm	1000-1120 µm
Example A	2.4	0.5	0.9	1.9	4.9	4.0	7.0
Example B	1.2	0.2	0.6	1.5	4.2	3.8	7.1
Example C	2.2	0.7	1.3	2.3	6.1	4.8	8.6
Example D	2.2	0.5	1.0	1.8	4.7	4.0	7.2

Sample	1120-1250 µm	1250-1400 µm	1400-1600 µm	1600-1700 µm	1700-2000 µm	> 2000 µm
Example A	8.0	8.9	15.3	7.4	22.7	16.1
Example B	8.6	9.8	15.6	7.1	22.2	18.1
Example C	10.6	11.0	14.5	6.6	19.2	12.1
Example D	7.7	8.0	15.1	6.4	22.6	18.8

2.Examples E and F: Preparation of a compacted gylcine having improved flowability and storage stability (compacted glycine having a low coarse content >2000µm)

Compaction Example E and F:

Speed of rotation of screw 17 to 19 rpm,

Speed of rotation of rollers 21 rpm, pressure at the pressure rollers 9.15 +/- 0.65 KN/cm

2 samples were taken and characterised: Example E and Example F

Bulk density, tapped density, Hausner factor, compressibility index:

(details on the measurement methods, see under Methods)

Sample	Bulk density (g/ml)	Tapped density (g/ml)	Hausner factor	Compres-sibility index (%)	Drying loss (%)
Example E	0.72	0.80	1.12	10.6	0.03
Example F	0.72	0.81	1.12	10.4	0.03

Particle distribution determined by tower sieving:

Figures in % by weight (details on the measurement method, see under Methods)

Sample	<500 µm	500-600 µm	600-710 µm	710-800 µm	800-900 µm	900-1000 µm
Example E	0.2	0.2	0.1	0.5	1.6	4.2
Example F	0.4	0.0	0.2	0.5	1.5	4.3

Sample	1000-1120 µm	1120-1250 µm	1250-1400 µm	1400-1600 µm	1600-1700 µm	1700-2000 µm	> 2000 µm
Example E	9.7	12.1	13.5	25.2	13.1	19.4	0.2
Example F	11.2	14.0	15.7	26.1	10.8	15.3	0.0

3. Comparisons 1 and 2: commercially available crystalline glycine

2 batches of a commercially available glycine are employed for comparative purposes (as prior art)

Comparison 1: Glycine cryst. suitable for use as excipient EMPROVE® exp Ph.Eur., BP, JP, USP Art.No. 1.00590.9025; batch: VP708290

Comparison 2: Glycine cryst. suitable for use as excipient EMPROVE® exp Ph.Eur., BP, JP, USP Art.No. 1.00590.9025; batch: VP709890

Bulk density, tapped density, Hausner factor, compressibility index:

(details on the measurement methods, see under Methods)

Sample	Bulk density (g/ml)	Tapped density (g/ml)	Hausner factor	Compressibility index (%)	Drying loss (%)
Comparison 1	1.01	1.15	1.14	12.5	0.02
Comparison 2	1.03	1.26	1.23	18.4	0.02

Although both comparisons exhibit a very low water content and also comparable bulk and tapped densities, they differ in the level of the Hausner factor and in the compressibility index (in accordance with Ph Eur 6th Edition, Table 2.9.36-2 "Scale of flowability", comparison 1 should be classified as "good" comparison 2 as "satisfactory") – in the visual assessment, comparison 2 already exhibits slight caking tendencies compared with comparison 1, but where the agglomerates can be destroyed by slight application of pressure.

Particle distribution determined by tower sieving:

Figures in % by weight (details on the measurement method, see under Methods)

Sample	<32 µm	32-50 µm	50-100 µm	100-150 µm	150-200 µm	200-250 µm	250-300 µm	300-355 µm
Comparison 1	0.0	0.5	1.5	5.3	6.0	8.6	12.9	8.5
Comparison 2	0.0	5.6	6.7	8.5	9.3	12.3	15.8	8.2

Sample	355-400 µm	400-500 µm	500-600 µm	600-710 µm	710-1000 µm	> 1000 µm
Comparison 1	8.2	17.0	15.9	8.6	7.0	0.0
Comparison 2	8.9	13.3	7.7	2.6	1.1	0.0

C) Results

Summary of the experimental results

1. Glycine (examples according to the invention and comparisons) exhibit an identical behaviour in the change in mass in the DVS; only from >90% RH is any increase in the mass evident at all, i.e. the material – irrespective of whether examples according to the invention or comparisons - is virtually non-hygroscopic up to 90% RH. The increases in weight at 98% RH even exhibit a slightly lower uptake of water for the rapidly caking comparisons 1 and 2 compared with the stable Examples A-F.
2. Changes in the drying losses after stress loading are not evident – all samples (examples according to the invention and comparisons) behave the same and exhibit no hygroscopicity
3. In spite of this missing tendency for the uptake of water, the samples surprisingly exhibit clear differences in their flow behaviour on storage under stress loading: whereas Examples A to F remain flowable, comparisons 1 and 2 cake after only a very short time and can no longer be removed in free-flowing form from the storage vessels – this even applies to comparison 1 with its very good initial value with respect to the flow behaviour.
4. Experiments and comparisons show that the compacted glycine with its particular particle size >700 µm remains flowable for significantly longer than the comparative material, even after storage under stress conditions, i.e. elevated temperature and atmospheric humidity.

5. The dissolution behaviour of Example A to F is virtually unchanged, even after storage, and has a sufficient rate for problem-free further processing

Experimental results in detail

1) Hygroscopicity (DVS)

Both Examples A to F according to the invention and comparisons 1 and 2 only exhibit an increase in weight (DVS) from a rel. humidity of >90%. At a rel. humidity of 98%, the compacted and non-lumping Examples A to E even have a tendency towards a slightly greater increase in weight than comparisons 1 and 2.

DVS measurement (dynamic vapor sorption):

	Relative humidity (%)										
	0.0	10.0	20.0	30.0	40.0	50.0	60.0	70.0	80.0	90.0	98.0
Example A	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.01	0.03	2.29
Example B	0.00	0.00	0.00	0.00	0.01	0.01	0.01	0.01	0.02	0.03	3.25
Example C	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.01	0.02	0.04	2.70
Example D	0.00	0.00	0.01	0.01	0.01	0.01	0.01	0.02	0.02	0.03	3.15
Example E	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.01	0.02	2.49
Example F	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.01	0.02	2.86
Comparison 1	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.01	0.01	0.01	1.83
Comparison 2	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.02	1.40

Increases in weight figures in "% by weight"

2) Drying losses after storage under stress conditions

Figures in % by weight

Storage condition: 25°C/60% RH open storage

Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example A	no storage takes place			
Example B	no storage takes place			
Example C	no storage takes place			
Example D	no storage takes place			
Example E	0.03	0.05	0.05	0.09
Example F	0.03	0.09	0.02	0.09
Comparison 1	0.02	0.07	0.01	0.04
Comparison 2	0.02	0.05	0.04	0.02

Storage condition: 25°C/60%RH closed storage

Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example A	no storage takes place			
Example B	no storage takes place			
Example C	no storage takes place			
Example D	no storage takes place			
Example E	0.03	0.06	0.03	0.08
Example F	0.03	0.08	0.06	0.10
Comparison 1	0.02	0.01	0.02	0.04
Comparison 2	0.02	0.05	0.03	0.02

Storage condition: 40°C/75%RH open storage

Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example A	0.02	-	0.01	0.02
Example B	0.04	-	0.04	< 0.01
Example C	0.05	-	< 0.01	0.01
Example D	0.04	-	< 0.01	< 0.01
Example E	0.03	0.06	0.03	0.08
Example F	0.03	0.04	0.04	0.07
Comparison 1	0.02	0.01	0.01	0.04
Comparison 2	0.02	0.04	0.03	0.03

Storage condition: 40°C/75%RH closed storage

Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example A	0.02	-	< 0.01	< 0.01
Example B	0.04	-	0.04	< 0.01
Example C	0.05	-	< 0.01	0.01
Example D	0.04	-	0.02	0.025
Example E	0.03	0.01	0.03	0.08
Example F	0.03	0.08	0.04	0.09
Comparison 1	0.02	0.03	0.02	0.04
Comparison 2	0.02	0.05	0.02	0.03

3) Changes in weight after storage under stress conditions

Storage condition: 25°C/60% RH open storage

Sample	Difference [g] after 1 week	Difference [g] after 2 weeks	Difference [g] after 7 weeks
Example A	no storage takes place		
Example B	no storage takes place		
Example C	no storage takes place		
Example D	no storage takes place		
Example E	0.01	-0.03	0.00
Example F	-0.02	0.03	0.00
Comparison 1	0.00	-0.01	0.04
Comparison 2	0.01	0.07	0.08

Storage condition: 25°C/60%RH closed storage

Sample	Difference [g] after 1 week	Difference [g] after 2 weeks	Difference [g] after 7 weeks
Example A	no storage takes place		
Example B	no storage takes place		
Example C	no storage takes place		
Example D	no storage takes place		
Example E	-0.01	-0.02	0.00
Example F	-0.01	-0.01	0.01
Comparison 1	0.00	-0.01	-0.02
Comparison 2	0.04	0.08	0.00

Storage condition: 40°C/75%RH open storage

Sample	Difference [g] after 1 week	Difference [g] after 2 weeks	Difference [g] after 7 weeks
Example A	-	0.01	0.00
Example B	-	0.00	-0.01
Example C	-	-0.01	-0.01
Example D	-	0.00	-0.02
Example E	0.04	0.00	0.04
Example F	-0.01	0.03	-0.02
Comparison 1	-0.02	0.03	0.00
Comparison 2	0.02	0.02	-0.04

Storage condition: 40°C/75%RH closed storage

Sample	Difference [g] after 1 week	Difference [g] after 2 weeks	Difference [g] after 7 weeks
Example A	-	-0.02	-0.03
Example B	-	-0.02	-0.03
Example C	-	0.01	-0.04
Example D	-	0.00	-0.04
Example E	0.07	-0.02	0.00
Example F	-0.03	-0.01	-0.02
Comparison 1	0.03	0.01	-0.06
Comparison 2	0.01	0.03	-0.01

4)

4) Changes in the dissolution rates after storage under stress conditions (figures in "seconds")

Storage condition: 25°C/60% RH open storage

Sample	Initial value /day of starting storage	after 1 week	after 2 weeks	after 7 weeks
Example E	318 ± 20	331 ± 13	343 ± 7	328 ± 19
Example F	316 ± 15	300 ± 7	341 ± 10	343 ± 20
Comparison 1	233 ±51	176 ± 14	194 ± 7	198 ± 6
Comparison 2	127 ± 11	181 ± 8	185 ±5	191 ± 9

Storage condition: 25°C/60%RH closed storage

Sample	Initial value /day of starting storage	after 1 week	after 2 weeks	after 7 weeks (
Example E	318 ± 20	328 ± 7	352 ± 8	358 ± 19
Example F	316 ± 15	301 ± 13	339 ± 6	363 ± 25
Comparison 1	233 ±51	176 ± 13	216 ± 6	207 ± 20
Comparison 2	127 ± 11	173 ± 6	209 ±5	196 ± 5

Storage condition: 40°C/75%RH open storage

Sample	Initial value /day of starting storage	after 1 week	after 2 weeks	after 7 weeks
Example A	358 ± 12	-	335 ± 11	313 ± 5
Example B	347 ± 11	-	340 ± 16	309 ± 12
Example C	339 ± 6	-	346 ± 18	324 ± 20
Example D	348 ± 5	-	332 ± 9	270 ± 10

Sample	Initial value / day of starting storage	after 1 week	after 2 weeks	after 7 weeks
Example E	318 ± 20	336 ± 12	326 ± 22	337 ± 10
Example F	316 ± 15	312 ± 18	332 ± 13	330 ± 12
Comparison 1	233 ±51	192 ± 9	202 ± 13	191 ± 17
Comparison 2	127 ± 11	182 ± 5	187 ±6	209 ± 12

Storage condition: 40°C/75%RH closed storage

Sample	Initial value / day of starting storage	after 1 week	after 2 weeks	after 7 weeks
Example A	358 ± 12	-	350 ± 23	297 ± 4
Example B	347 ± 11	-	332 ± 12	304 ± 9
Example C	339 ± 6	-	326 ± 10	299 ± 6
Example D	348 ± 5	-	319 ± 11	290 ± 1
Example E	318 ± 20	322 ± 8	343 ± 5	343 ± 14
Example F	316 ± 15	316 ± 7	350 ± 15	342 ± 12
Comparison 1	233 ±51	177 ± 6	215 ± 8	203 ± 4
Comparison 2	127 ± 11	201 ± 17	193 ±10	215 ± 9

5) Clumping behaviour after storage under stress conditions (visual description)

"Free-flowing" = the glycine flows freely without input of force – agglomerates are not visually evident.
"Lumpy" = the glycine exhibits individual relatively large agglomerates (and encrustations (of diameter about 1 cm), but is still free-flowing.
"Slightly caked" = the glycine is solid; however, it can be converted back into the free-flowing state by slight input of force (gentle poking with a glass rod or spatula, hitting or shaking).
"Strongly caked" = the glycine is solid; strong input of force (vigorous poking with a glass rod or spatula) is necessary in order to break up the glycine again - however, the glycine is still strongly agglomerated after this (chunk formation) and is not free-flowing.

Storage condition: 25°C/60% RH open storage

Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example E	free-flowing	free-flowing	free-flowing	free-flowing
Example F	free-flowing	free-flowing	free-flowing	free-flowing
Comparison 1	free-flowing	slightly caked	slightly caked	slightly caked
Comparison 2	lumpy	strongly caked	strongly caked	strongly caked

Storage condition: 25°C/60%RH closed storage

Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example E	free-flowing	free-flowing	free-flowing	free-flowing
Example F	free-flowing	free-flowing	free-flowing	free-flowing
Comparison 1	free-flowing	lumpy	free-flowing	slightly caked
Comparison 2	lumpy	strongly caked	strongly caked	strongly caked

Storage condition: 40°C/75%RH open storage

Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example A	free-flowing	-	slightly caked	slightly caked
Example B	free-flowing	-	slightly caked	slightly caked
Example C	free-flowing	-	slightly caked	slightly caked
Example D	free-flowing	-	slightly caked	slightly caked
Example E	free-flowing	slightly caked	slightly caked	slightly caked
Example F	free-flowing	slightly caked	slightly caked	slightly caked
Comparison 1	free-flowing	strongly caked	strongly caked	strongly caked
Comparison 2	lumpy	strongly caked	strongly caked	strongly caked

Storage condition: 40°C/75%RH closed storage

Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example A	free-flowing	-	free-flowing	free-flowing (weak agglomerates)
Example B	free-flowing	-	free-flowing	free-flowing
Example C	free-flowing	-	free-flowing	free-flowing
Example D	free-flowing	-	free-flowing	free-flowing
Example E	free-flowing	free-flowing	free-flowing	free-flowing
Example F	free-flowing	free-flowing	free-flowing	free-flowing
Comparison 1	free-flowing	strongly caked	strongly caked	strongly caked
Comparison 2	lumpy	strongly caked	strongly caked	strongly caked

6) Changes in the flow behaviour (measured as change in the "avalanche angle" in the Revolution powder analyser) after storage under stress conditions

Storage condition: 25°C/60% RH open storage

	Avalanche angle [°]			
Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example E	43.6	42.5	41.8	42.1
Example F	45.6	43.2	41.9	42.2
Comparison 1	39.4	40.8	39.5	40.2
Comparison 2	65.6	n.m.	n.m.	n.m.

Storage condition: 25°C/60%RH closed storage

	Avalanche angle [°]			
Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example E	43.6	42.6	41.8	42.1
Example F	45.6	42.8	43.2	42.3
Comparison 1	39.4	41.8	39.6	40.5
Comparison 2	65.6	n.m.	n.m.	n.m.

Storage condition: 40°C/75%RH open storage

	Avalanche angle [°]			
Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example E	43.6	43.8	43.2	43.5
Example F	45.6	43.7	42.7	42.6
Comparison 1	39.4	n.m.	n.m.	n.m.
Comparison 2	65.6	n.m.	n.m.	n.m.

Storage condition: 40°C/75%RH closed storage

	Avalanche angle [°]			
Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example E	43.6	42.8	42.8	42.9
Example F	45.6	43.4	42.4	42.8
Comparison 1	39.4	n.m.	n.m.	n.m.
Comparison 2	65.6	n.m.	n.m.	n.m.

n.m. = not measurable since the material has already caked too strongly in order to be removed from the vessel (too many agglomerates are present or the material has to be poked out of the dish/bottle)

7) Changes in the flow behaviour (measured as change in the "break energy" in the Revolution powder analyser) after storage under stress conditions

Storage condition: 25°C/60% RH open storage

	Break energy [mJ]			
Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example E	28.4	28.1	27.7	27.9
Example F	28.8	28.6	28.3	28.2
Comparison 1	27.4	27.5	27.2	27.5
Comparison 2	33.5	n.m.	n.m.	n.m.

Storage condition: 25°C/60%RH closed storage

	Break energy [mJ]			
Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example E	28.4	28.1	27.7	27.9
Example F	28.8	28.5	28.6	27.9
Comparison 1	27.4	28.0	27.1	27.4
Comparison 2	33.5	n.m.	n.m.	n.m.

Storage condition: 40°C/75%RH open storage

	Break energy [mJ]			
Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example E	28.4	28.7	28.0	28.0
Example F	28.8	28.7	28.3	28.1
Comparison 1	27.4	n.m.	n.m.	n.m.
Comparison 2	33.5	n.m.	n.m.	n.m.

Storage condition: 40°C/75%RH closed storage

	Break energy [mJ]			
Sample	Initial value	after 1 week	after 2 weeks	after 7 weeks
Example E	28.4	28.3	28.4	28.2
Example F	28.8	28.9	28.0	28.5
Comparison 1	27.4	n.m.	n.m.	n.m.
Comparison 2	33.5	n.m.	n.m.	n.m.

n.m. = not measurable since the material has already caked too strongly in order to be removed from the vessel (too many agglomerates are present or the material has to be poked out of the dish/bottle)

Claims

1. Glycine granules where at least 75% (w/w) of the glycine granules have a particle size of at least 0.7 mm.

2. Glycine granules according to Claim 1, characterised in that at least 80% (w/w) of the glycine granules have a particle size of at least 0.8 mm.

3. Glycine granules according to Claim 1 or 2, characterised in that the glycine granules have a bulk density of less than or equal to 0.9 g/ml.

4. Glycine granules according to one or more of Claims 1 to 3, characterized in that the glycine granules are flowable after storage for 3 months in a closed container at room temperature.

5. Glycine granules according to Claim 1, which can be prepared by compaction.

6. Glycine granules according to Claim 5, characterised in that the preparation is carried out by
a) provision of glycine powder
b) compaction of the glycine powder from step a) in a roller compactor.

7. Glycine granules according to Claim 5 or 6, characterised in that the pressing force of the roller compactor in step b) is between 1 and 50 KN/cm of roller width.

8. Glycine according to one or more of Claims 5 to 7, characterised in that the preparation is carried out by
a) provision of glycine powder
b) compaction of the glycine powder from step a) in a roller compactor, giving a compact which comprises particles having a particle size above 0.7 mm
c) At least partial recycling of glycine particles from the compact obtained in step b) that have a particle size smaller than 0.7 mm to the glycine powder provided in step a).

9. Glycine according to one or more of Claims 5 to 8, characterised in that the recycling in step c) is carried out by crushing the compacts and classification by particle size, where at least some of the glycine particles that have a particle size smaller than 0.7 mm are recycled to the glycine powder provided in step a).

10. Process for the preparation of glycine granules where at least 75% (w/w) of the glycine granules have a particle size of at least 0.7 mm, by
a) provision of glycine powder
b) compaction of the glycine powder from step a).

11. Process according to Claim 10, characterised in that the preparation is carried out by
a) provision of glycine powder

b) compaction of the glycine powder from step a) in a roller compactor, giving a compact which comprises particles having a particle size above 0.7 mm
c) At least partial recycling of glycine particles from the compact obtained in step b) that have a particle size smaller than 0.7 mm to the glycine powder provided in step a).

12. Process according to Claim 10 or 11, characterised in that the pressing force of the roller compactor in step b) is between 1 and 50 KN/cm of roller width.

13. Process according to one or more of Claims 10 to 12, characterised in that the recycling in step c) is carried out by crushing the compacts and classification by particle size, where at least some of the glycine particles that have a particle size smaller than 0.7 mm are recycled to the glycine powder provided in step a).

14. Use of glycine granules where at least 75% (w/w) of the glycine granules have a particle size of at least 0.7 mm, for the preparation of nutrient media for medical applications or in biotechnology.

15. Use according to Claim 14, characterised in that the glycine granules have been compacted.