
**Plastics — Preparation of PVC pastes for
test purposes — Planetary-mixer method**

*Plastiques — Préparation des pâtes PVC pour essais — Méthode utilisant
un mélangeur planétaire*

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 4612 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 4612:1979), which has been modified to make it suitable for the preparation of pastes of various compositions and to include different types of planetary mixer.

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Printed in Switzerland

Plastics — Preparation of PVC pastes for test purposes — Planetary-mixer method

1 Scope

This International Standard specifies two methods, A and B, for the preparation of pastes (also known as plastisols) from appropriate PVC resins, plasticizers and other ingredients using a planetary mixing process.

Both method A and method B can be used to prepare pastes of any composition. Method A (single-speed) is particularly applicable to resins prone to heat build-up during paste preparation, while method B (two-speed) might be preferred for repetitive work, e.g. for process control during resin manufacture, because of its shorter mixing time.

Such pastes may be used for a variety of test purposes, including the determination of rheological properties for resin designation and specification.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments or revisions apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing*.

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

3.1

paste

plastisol

a homogeneous dispersion of PVC resin in an organic liquid

3.2

planetary mixer

a mixer in which the beater or stirrer not only rotates about an axis within the mixing vessel, but the axis of rotation itself also describes a circular path

4 Principle

Liquid plasticizer, PVC resin powder and other appropriate ingredients, in liquid, paste or powder form, are combined in a mixer where the shearing action causes the powder(s) to disperse in the liquid(s).

The paste-making process involves the partial solvation of the resin powder by the plasticizer, and hence the "dispersion" will remain in liquid form for prolonged periods, i.e. the powder does not readily settle out.

5 Apparatus and materials

5.1 Planetary mixer, having at least the key features shown in Figure 1, and with a mixing-bowl/beater configuration similar to one of those illustrated in Figure 2.

The mixing bowl shall be made of stainless steel and may be fitted with a jacket for the circulation of cooling water. The bowl will have a recommended filling level, stated by the manufacturer. This shall allow the efficient mixing of 700 g to 1 200 g of charge, made up of the relevant proportions of resin, plasticizer and any other ingredients.

The motor specified shall have sufficient power to maintain the speed of rotation of the beater around the axis of the bowl at $60 \text{ min}^{-1} \pm 5 \text{ min}^{-1}$ and additionally, if method B is used, at $120 \text{ min}^{-1} \pm 10 \text{ min}^{-1}$ with the most viscous paste that it is intended to mix. There will be a fixed relationship between the speed of rotation of the beater around the axis of the bowl and the speed of rotation of the beater around its own axis that will have been set by the manufacturer of the particular mixer. It shall approximate to the ratio 3:7, i.e. when the speed of rotation of the beater around the axis of the bowl is set at 60 min^{-1} , the speed of rotation around its own axis will be approximately 140 min^{-1} .

The mixer may be provided with a rotating wiper to scrape the inside of the bowl with a blade made of a material that is resistant to the liquid(s) used to make the paste.

The mixer may also have facilities for applying a vacuum to the contents of the bowl, both during and after mixing.

5.2 Controlled-atmosphere cabinet or room, capable of maintaining $23 \text{ °C} \pm 2 \text{ °C}$ and $(50 \pm 5) \%$ relative humidity.

5.3 Balance, accurate to 0,5 g.

5.4 Timing device, which indicates seconds.

5.5 Beakers, of different capacities, for containing the test specimens of paste. The beakers shall be big enough to avoid paste overflowing during vacuum de-aeration of the paste.

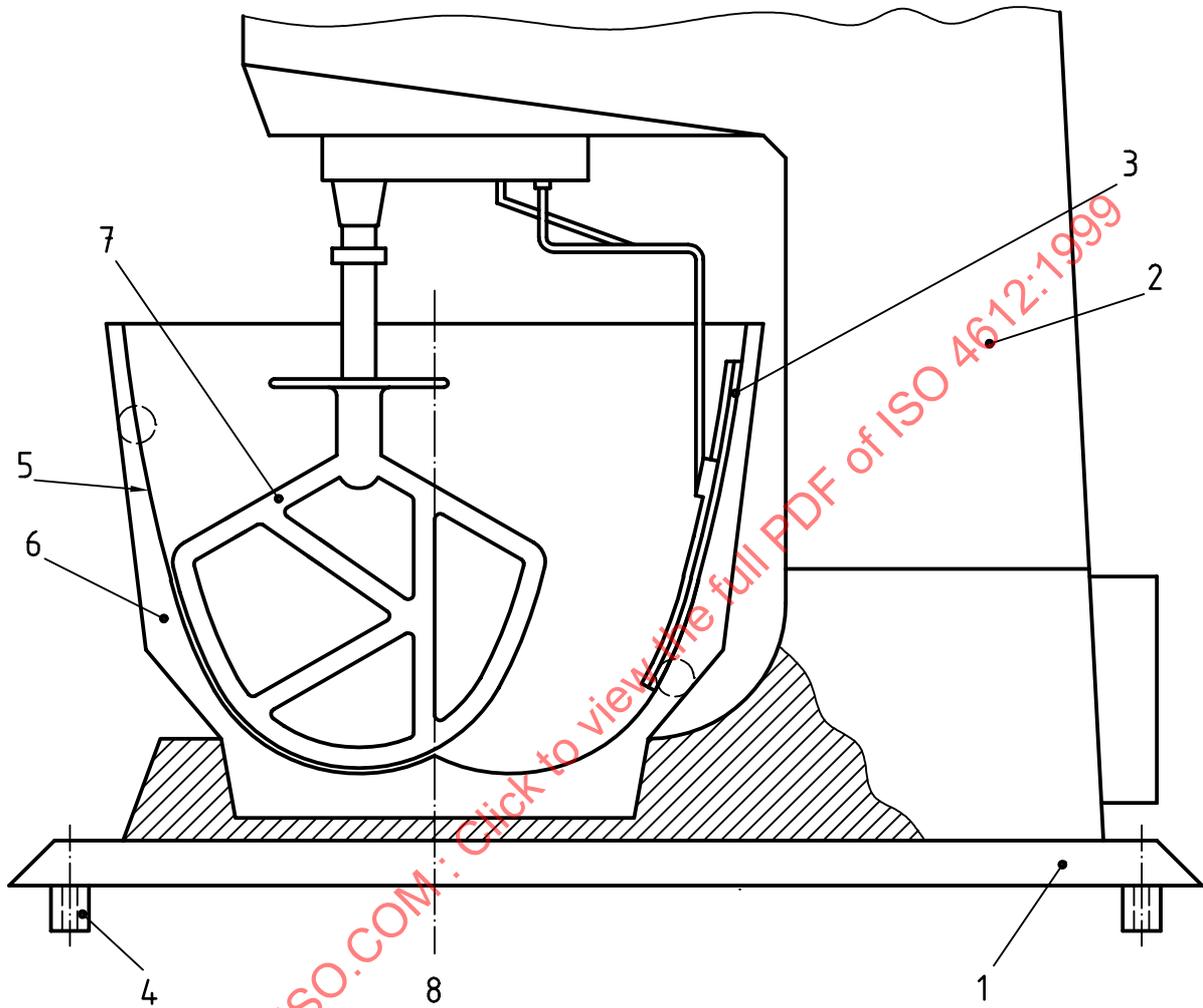
5.6 Vacuum pump, desiccator, etc., to enable the de-aeration of pastes at a partial vacuum of about $2 \times 10^3 \text{ Pa}$.

5.7 Palette knife.

5.8 Plasticizer.

6 Resin sample and conditioning

The resin used to make the paste shall be free from agglomerates and shall be conditioned at $23 \text{ °C} \pm 2 \text{ °C}$ and $(50 \pm 5) \%$ relative humidity, in accordance with ISO 291, for 24 h prior to use.



Key

- 1 Base
- 2 Mixer
- 3 Optional wiper or scraper, rotating to clean inside of bowl
- 4 Feet
- 5 Stainless-steel bowl
- 6 Optional jacket for circulation of cooling water
- 7 Beater
- 8 Main axis of rotation

Figure 1 — Planetary mixer

7 Preparation of paste

7.1 Method A

Weigh 100 % of the calculated quantity of resin into the mixer bowl followed by 100 % of the calculated quantity of plasticizer. The amount of plasticizer is determined both by the paste formulation and by the total quantity of resin, plasticizer and any other ingredients which is necessary to fill the mixer bowl to the level for "efficient" mixing (see 5.1). Any other powder-form ingredients should preferably be added as part of the resin system, while other liquid ingredients should be included with the plasticizer. Depending on the amount involved, ingredients in paste form can be added with the powders or with the liquids.

Using the palette knife, hand-mix the plasticizer and resin until no free plasticizer can be seen. Scrape the knife clean with the mixer beater to ensure no material is lost. Locate the mixer bowl on the mixer frame, attach the beater to the shaft and raise the bowl or lower the mixer with its beater to the operating position.

Start the timer and the mixer simultaneously, with the beater rotating at $60 \text{ min}^{-1} \pm 5 \text{ min}^{-1}$ around the axis of the bowl. After $1 \text{ min} \pm 10 \text{ s}$, stop the mixer and timer, take out the bowl and use the palette knife to scrape down the sides of the bowl and the beater. Clean the knife on the beater, put the bowl back and restart the mixer and timer. After a further $5 \text{ min} \pm 15 \text{ s}$, repeat the scrape-down procedure described earlier and then continue mixing until the total actual mixing time reaches $30 \text{ min} \pm 30 \text{ s}$.

If the mixer is fitted with a rotating scraper for the bowl, it is not necessary to stop mixing in order to scrape down the sides of the bowl, and mixing shall be allowed to continue uninterrupted for the full $30 \text{ min} \pm 30 \text{ s}$. If, however, material is noticed adhering to the beater, use the procedure described for mixers having no rotating scraper.

Stop the mixer and immediately check the temperature of the paste. If the temperature is $> 35 \text{ }^\circ\text{C}$, prepare a new paste either using more plasticizer or, if the mixer bowl is fitted with a cooling jacket, passing cooling water through the jacket.

Transfer the paste to suitable beakers. If lumps are visible, prepare a new batch of paste. If not, de-aerate the paste under a partial vacuum of about $2 \times 10^3 \text{ Pa}$ and maintain this vacuum for about 5 min after the foam has been observed to collapse. If the mixer has the necessary facilities, the de-aeration procedure may be carried out in the mixer bowl.

Cool and store the paste at $23 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ and $(50 \pm 5) \%$ relative humidity with the beaker covered to prevent accidental contamination.

Take care throughout these procedures to avoid spillage and spattering.

7.2 Method B

Weigh 100 % of the calculated quantity of resin into the mixer bowl followed by 100 % of the calculated quantity of plasticizer. The amount of plasticizer is determined both by the paste formulation and by the total quantity of resin, plasticizer and any other ingredients which is necessary to fill the mixer bowl to the level for "efficient" mixing (see 5.1). Any other powder-form ingredients should preferably be added as part of the resin system, while other liquid ingredients should be included with the plasticizer. Depending on the amount involved, ingredients in paste form can be added with the powders or with the liquids.

Using the palette knife, hand-mix the plasticizer and resin until no free plasticizer can be seen. Scrape the knife clean with the mixer beater to ensure no material is lost. Locate the mixer bowl on the mixer frame, attach the beater to the shaft and raise the bowl or lower the mixer with its beater to the operating position.

Start the timer and the mixer simultaneously, with the beater rotating at $60 \text{ min}^{-1} \pm 5 \text{ min}^{-1}$ around the axis of the bowl. After $2 \text{ min} \pm 10 \text{ s}$, stop the mixer and timer, take out the bowl and use the palette knife to scrape down the sides of the bowl and the beater. Clean the knife on the beater, put the bowl back and restart the mixer and timer, with the beater now rotating at $120 \text{ min}^{-1} \pm 10 \text{ min}^{-1}$. Continue mixing until the total actual mixing time reaches $20 \text{ min} \pm 30 \text{ s}$.

If the mixer is provided with a rotating scraper for the bowl, it is not necessary to stop mixing in order to scrape down the sides of the bowl, and mixing shall be allowed to continue for the full $20 \text{ min} \pm 30 \text{ s}$, stopping only briefly after $2 \text{ min} \pm 10 \text{ s}$, if necessary, to increase the beater speed from $60 \text{ min}^{-1} \pm 5 \text{ min}^{-1}$ to $120 \text{ min}^{-1} \pm 10 \text{ min}^{-1}$. If, however, material is noticed adhering to the beater, use the procedure described for mixers having no rotating scraper.

Stop the mixer and immediately check the temperature of the paste. If the temperature is $> 35 \text{ }^\circ\text{C}$, prepare a new paste either using more plasticizer or, if the mixer bowl is fitted with a cooling jacket, passing cooling water through the jacket. Note that changing to method A may help to achieve a lower final paste temperature.

Transfer the paste to suitable beakers. If lumps are visible, prepare a new batch of paste. If not, de-aerate the paste under a partial vacuum of about $2 \times 10^3 \text{ Pa}$ and maintain this vacuum for about 5 min after the foam has been observed to collapse. If the mixer has the necessary facilities, the de-aeration procedure may be carried out in the mixer bowl.

Cool and store the paste at $23 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ and $(50 \pm 5) \%$ relative humidity with the beaker covered to prevent accidental contamination.

Take care throughout these procedures to avoid spillage and spattering.

8 Paste-preparation report

The report shall include the following information:

- a) a reference to this International Standard;
- b) the paste formulation;
- c) the temperature of the paste on completion of mixing;
- d) the date of preparation of the paste;
- e) any deviation from this International Standard.