

TECHNICAL INFORMATION GRANODINE 20

1. INTRODUCTION

Granodine 20 is a concentrated liquid chemical, which when diluted with water, forms adherent zinc phosphate coatings on steel and zinc surfaces immersed in the bath.

The coatings produced are very fine, tight and hard, giving excellent adhesion; humidity and corrosion resistance to subsequent paint coatings.

The **Granodine 20** bath produces negligible sludge formation and scale build-up on the heating coils. **Granodine 20** can be preceded by strong alkali cleaner or acid pickle without the need for refining additives in the rinse stage.

2. MAKE-UP AND OPERATION

Granodine 20 is usually made up at 30 litres per 1,000 litres of bath, and the working bath is controlled as indicated by two titration tests.

The normal bath temperature is 80-95°C and the immersion time is usually two minutes, with up to five minutes for heavy coatings. Refer to Section 9 for a process variation allowing bath temperatures of 60-70°C.

PROCESS SEQUENCE

1. Clean
2. Hot Water Rinse
3. Coat
4. Cold Water Rinse
5. Passivation

Coatings are then usually dried and painted.

3. EQUIPMENT

The tank for **Granodine 20** may be of stainless steel (preferably Type 316) or of heavy mild steel. Other stages require only mild steel.

Heated tanks should be equipped with steam plate coils and side heating (for more even temperature distribution) or other suitable heat sources.

Crates, baskets, barrels, conveyors, etc. may be of mild steel.

Detailed recommendations on equipment and specific process sequences are available from Henkel technical representatives.

4. BATH MAKE-UP AND CONTROL

Make-Up

For each 1,000 litres of water in the bath; add with stirring:-	
Water	900 litres
Granodine 20	30 litres

Mix and make-up to volume (1,000 litres) with more water. Heat to 85°C and add about 1 kg of steel wool for each 1,000 litres of bath.

Allow the steel wool to dissolve for 50 minutes (to achieve a ferrous iron content of 0.1%).

Remove the remains of the steel wool, and the bath is then ready for work. At this point, the Free Acid should be about 2.7-3.0 mL.

Control Points (for normal conditions)

Total Acid Titration	20 - 24 mL
Ferrous Iron Titration	3.6 - 16.0 mL
Temperature	80-95°C
Immersion Time	2-3 minutes

5. SURFACE PREPARATION

Surfaces to be coated with **Granodine 20** must be free of scale, rust, oil, grease and shop dirt. They may be cleaned with a hot solution of **Parco Cleaner** (e.g. **Parco Cleaner 415**, or **1523**) trichlorethylene vapour degreasing, or other suitable method.

The **Parco Cleaner** stages are usually followed by two 30 second or one 60 second rinse at 60-70°C with continuous overflow to avoid the build-up of contaminants.

6. TESTING AND BATH MAINTENANCE

General

The **Granodine 20** bath is controlled by Total Acid and Ferrous Iron titrations.

Total Acid level is tested regularly, and determines the required amount of **Granodine 20** to replenish the bath.

Ferrous Iron is an important constituent of the bath, but it may build-up to excessive levels during heavy loading of the bath. It should be measured occasionally or whenever coating problems are encountered.

Total Acid Titration

- (a) Pipette a 10 mL sample into a conical flask.
- (b) Add 6-10 drops of phenolphthalein indicator, and 50-100 mL of water.
- (c) Titrate slowly with 0.1N sodium hydroxide until a permanent faint pink colour is obtained.
- (d) Record the number of millilitres (mL) of 0.1N sodium hydroxide as Total Acid.
- (e) Add 1.4 litres of **Granodine 20** per 1,000 litres of bath to raise the Total Acid by 1.0.

Ferrous Iron Titration

- (a) Pipette a 10 mL sample into a conical flask.
- (b) Add 10-20 drops of 50% Sulphuric acid.
- (c) Titrate slowly with 0.042N potassium permanganate to a permanent faint pink colour.
- (d) Record the number of millilitres (mL) of 0.042N potassium permanganate as Ferrous Iron titration. (1 mL of 0.042N potassium permanganate is equivalent to 0.23% ferrous iron.)

If the Ferrous Iron Titration is over 16 mL: dump an appropriate portion of the bath, to reduce the Ferrous Iron titration to 16 mL or less; and replenish with **Granodine 20** and water (in the suggested make-up proportions) to restore the Total Acid titration.

7. OPERATIONAL RECOMMENDATIONS

Variations in operating conditions are often required to obtain optimum results, due to specific plant, work and production changes. Your Henkel technical representative should be contacted for detailed recommendations.

If the **Granodine 20** bath is allowed to operate above 95°C, high chemical cost and excessive sludging will result.

OPERATIONAL RECOMMENDATIONS Cont..

If the bath is allowed to operate below 80°C, or at too low a Total Acid; thin sparse coatings will result. Refer Section 9 for details of the lower temperature **Accelerator** Process variation.

8. AFTER TREATMENT

Cold Water Rinse

After immersion in the **Granodine 20** bath, the work should be rinsed for 30 seconds (min.) in a cold water tank, with continuous overflow to avoid the build-up of contaminants.

DEOXYLYTE* Passivation

The work is then rinsed for 30 seconds with clean, salt-free water containing **Deoxylyte** (for maximum corrosion resistance.) This stage may be operated hot to facilitate drying, and for some work, avoid the need for an oven dry off stage.

Drying

The work should then be dried as soon as possible in an indirect fired oven, or by other means which will not contaminate the coating with fumes, or partly burnt gases or oil.

Solution trapped in cavities should be removed by suction, or blowing etc. before the work enters the oven.

Low oven temperatures (i.e. 110°C or less) are preferred to maintain the phosphate coating in optimum condition.

9. LOWER TEMPERATURE PROCESS VARIATION (Using **Accelerator 131**)

Summary

Where an operating temperature of 80°C cannot be maintained in the **Granodine 20** bath; this process variation, operating at 60-70°C by adding a second chemical, **Accelerator 131**, should be used. This method increases the amount of sludge formed, and requires more operator control.

No other changes are required, nor result from the use of **Accelerator 131**, so the recommendations for cleaning, after treatment, **Granodine 20** concentrations, Total Acid and permanganate titrations, etc. apply also to this process variation.

Details of Process Variations

(a) Bath Make-up

In addition to the **Granodine 20**: about 10 minutes prior to processing work; add sufficient **Accelerator 131** to obtain a pale mauve to blue colour on Starch-Iodine paper (See Bath Control below.) This will normally require approximately 1 litre per 1,000 litres of bath.

(b) Testing

Accelerator level should be checked hourly by use of Starch-Iodine paper.

The colour of a strip of test paper, after being dipped in the bath, indicates the **Accelerator** level as follows:

Colour of Test Paper	Accelerator Level
Remains white	Too low
Pale mauve to blue	Correct
Blue-black to black	Too much

Accelerator Titration

For critical applications the following test may be required for more accurate control of the **Accelerator** level.

To avoid unnecessary titration, adjust the **Accelerator** level in the bath within the Starch-Iodine test paper, and then proceed as follows:

- (a) Pipette a 10 mL sample into a conical flask.
- (b) Add 10-20 drops of 50% sulphuric acid.
- (c) Titrate slowly with 0.042N potassium permanganate solution until a faint pink colour persists for 15 seconds after the addition of one drop.
- (d) Record the number of millilitres (mL) of 0.042N potassium permanganate used as the **Accelerator** titration.
- (e) Add 140 mL of **Accelerator 131** per 1,000 litres of bath to raise the **Accelerator** titration by 2.3 mL.

The **Accelerator** titration is normally kept between 9.5 and 29 mL by frequent small additions of **Accelerator 131**.

Note: The use of **Accelerator 131** eliminates any ferrous iron present in the bath, and consequently the need for the ferrous iron titration.)

10. PLANT MAINTENANCE

Water rinse stages are ideally discarded every day and made up afresh.

The cleaner stage should be skimmed to keep the surface clean and the bath discarded when excessive soil accumulates in the bath.

The **Granodine 20** bath during operating slowly generates a sludge which settles on the bottom of the tank, and forms scale on the heating coils. Both should be removed when they build up to a level which reduces the efficiency of the operation.

11. HANDLING PRECAUTIONS

Granodine 20 is a concentrated acidic chemical. Gloves, goggles and protective clothing should be worn for handling. If splashed on skin, or in eyes, wash it off immediately with soap and copious amounts of water, and obtain medical attention.

HENKEL PRODUCT REFERENCES

**Granodine 20
Accelerator 131**

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