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Use of Inhibitors for Scale Control in Brine-Producing Gas and Oil Wells

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ABSTRACT

Field and laboratory work sponsored by the Gas Research Institute (GRI) and the Department of Energy (DOE) have shown that calcium-carbonate scale formation in waters produced with natural gas and oil can be prevented by injection of phosphonate inhibitor into the formation, even if the formation is sandstone without calcite binding material. Inhibitor squeeze jobs have been carried out on DOE's geopressured-geothermal Gladys McCall brine-gas well and GRI's co-production wells in the Hitchcock field. Following the inhibitor squeeze on Gladys McCall, the well produced over five million barrels of water at a rate of approximately 30,000 BPD without calcium-carbonate scaling. Before the inhibitor squeeze, the well could not be produced above 15,000 BPD without significant scale formation. In the GRI brine-gas co-production field tests, inhibitor squeezes have been used to successfully prevent scaling.

Laboratory work has been conducted to determine what types of oil field waters are subject to scaling. This research has led to the development of a saturation index and accompanying nomographs which allow prediction of when scale will develop into a problem in brine production.

Core samples from both fields were used in laboratory studies and analytical methods to analyze inhibitors in brine at a low levels were extended. A complete history of field developments and the laboratory backup experiments is included in this paper.

References and illustrations at end of paper.

INTRODUCTION

Progress has been made toward controlling scale formation from brines often associated with geopressured energy production, co-production wells, and oil wells which make large amounts of water. As brine flows out of the formation and up the well, the pressure drops. This pressure drop causes dissolved carbon dioxide, CO_2 to go out of solution, which increases the solution pH. The pH rise causes aqueous bicarbonate, HCO_3^- to be converted to carbonate, CO_3^{2-} , which tends to initiate calcium carbonate, CaCO_3 , precipitation either in the formation pore throats near the well bore, on the production tubing walls or in surface handling equipment. Four scale control options include:

1. Limiting production so that the drop in pressure is not sufficient to induce precipitation (see below for details).
2. Injection of trace concentration of inhibitors in the surface equipment.
3. Injection of trace concentrations of inhibitors downhole via a small diameter treat string or down the annulus; and
4. squeezing inhibitor into the formation in such a manner that the inhibitor will be slowly released when production commences.

The first option, reduced production, generally entails an unacceptable loss in revenue. Injection of inhibitors into the surface equipment, option 2, does not protect the production tubing, and installation of a downhole treat string, option 3, is often prohibitively expensive. The last option, an inhibitor squeeze, can protect the near well bore formation, the production tubing, and the surface equipment. Successful inhibitor squeeze jobs have been

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carried out on DOE's geopressured-geothermal Gladys McCall brine-gas well near Grand Chanier, La, and GRI's co-production wells in the Hitchcock field near Galveston, Tx, and will be presented. The producing formation of the Gladys McCall well was secondary quartz cemented and contained no calcite, which made a successful inhibitor squeeze design considerably more complicated. Previous laboratory work has led to the development of a method to predict when scale will begin to form and how little inhibitor might be needed to prevent scale.

In this paper we will first discuss the field applications and results of phosphonate inhibitor squeezes to prevent formation of CaCO_3 scale. This will be followed by a description of laboratory experiments and theoretical considerations that led to the development of the inhibitor squeeze techniques employed.

FIELD EXPERIMENTS GLADYS MCCALL

The first attempts at phosphonate inhibitor squeezes at Gladys McCall were unsuccessful since the inhibitor could not be pumped into the formation. There appeared to be two possible causes for this: (1) Poor surface water quality with high iron and calcium content and (2) Interaction of the phosphonate with formation brine which contained 4000 PPM Ca ion (Table 1). The first of these problems could be handled by using good quality water which had passed through an ion exchange and a good filtering unit to remove any iron hydroxide. The second problem could be handled by using a brine spacer ahead of the inhibitor to force the calcium containing formation water away from the well bore. Based on these conclusions, the inhibitor pill was designed to be injected as follows:

1. 300 B of 15% NaCl spacer
2. 100 B of 3% inhibitor in 15% NaCl (the inhibitor was nitrilotri(methylene phosphonic) acid from Champion Chemical Co., Houston, Tx)
3. 100 B of 15% NaCl spacer
4. 100 B of 10% CaCl_2 overflush
5. 500 B of 15% NaCl into the formation as a pusher
6. The well was to be shut in for 24 hrs. to allow reaction

In fact, 6% inhibitor was used (step 2). Everything went well with the treatment at first. Pumping rate was held at 2 BPM with only a slight pressure increase over original shut in wellhead pressure. When the 100 B of inhibitor hit the formation, the pressure built up rapidly and the pump rate was reduced to 1 BPM to keep from exceeding fracturing pressure. The 100 B of inhibitor and 100 B of NaCl spacer was squeezed away at 1 BPM.

When the CaCl_2 overflush hit the formation, the pressure built up rapidly to the pre-set limit. Pump rate was reduced to 0.2 BPM and the 100 B of CaCl_2 solution was slowly pumped away. This was followed by approximately 25 B of 15% NaCl

shut in since all of the inhibitor was in the formation and had been exposed both to calcium from the formation brine and the CaCl_2 overflush as evidenced by the pressure increases.

After 24 hours, the well was allowed to flow back at 100 BPM. Brine samples were taken every 10 barrels during the flow back of the pill and periodically thereafter. These samples were analyzed for numerous elements, in addition to the inhibitor itself (Figure 1). It was found that magnesium was the most distinctive tracer for the formation brine although the early buildup (around 600 barrels of returns) could be due to impurities in the 15% NaCl and some interaction with the phosphonate. By the time 1200 barrels of brine had been produced, Mg, Ca, Na and K had stabilized to their original concentrations in formation brine. About 70% of the inhibitor flowed back with the first few thousand barrels of brine production. The remaining inhibitor was slowly released over the next six months. The concentration of the inhibitor dropped to about 0.1 to 0.2 mg/l within a few weeks and remained there until the well was shut in for repair and resqueeze in January, 1986. This corresponded to about 70% of the inhibitor remaining in the formation. In order to measure such low inhibitor concentrations in field brine it was necessary to modify standard colorimetric phosphonate procedures which will be published elsewhere. Prior to the inhibitor squeeze, production was limited to about 15,000 BPD in order to avoid scale formation. This severely curtailed gas production. After the squeeze it was possible to increase the production rate to about 30,000 BPD, still without scale formation in the production tubing. During this period about 25 SCF of natural gas was separated and sold per barrel of brine produced. At about four months into production, a light scale was observed in the final filters before the disposal well. This was eliminated by addition of 0.25 mg/l of inhibitor downstream of the choke. After six months of production no indication of scale formation in the production tubing was found, and when the well was shut in, the high pressure side of the choke was observed to be scale free. Thus, the phosphonate inhibitor squeeze had protected the tubing for the production of over five million barrels of brine.

Upon analysis of the breakthrough data of the flowback curves from the first squeeze job, it was concluded that the calcium in the formation brine could be used to form the calcium phosphonate in situ if sufficient mixing could be obtained in a large pill which was pumped a considerable distance from the well bore. This would avoid the pressure increase observed when the calcium of the CaCl_2 overflush hit the formation containing the pill and would greatly simplify the overall operation of the pill application.

Based on the observations from the behavior of the first successful phosphonate inhibitor squeeze, a simplified pill procedure was designed as follows:

1. Pump a 100 barrels preflush of 10% NaCl to push most of the Calcium ion in the reservoir

2. Use 100 barrels of pH neutral 3% (NH₄) phosphonate inhibitor salt in 10% NaCl solution which will react with calcium from formation water.
3. Overflush with 900 barrels of 10% NaCl followed by 300 barrels of oil field brine to clean the tubing.
4. Shut in for 36 hours and bring back slowly at 100 barrels per hour.
5. After 2 days resume flow of 30,000 barrels brine per day.

Since these fluids had approximately the same density as the formation brine and viscosities were low, the injection rate after preflush was chosen as 6 barrels per minute.

An oxygen scavenger (NH₄)HSO₃ and an iron chelating agent (EDTA) was added to all of the brine to prevent formation of iron hydroxide and a 2-1/2 micron filter was used just upstream of the pump suction. In addition, 100 millicuries of I 131 was mixed with the phosphonate pill as a tracer. The pressure curve for the injection is given in Figure 2. No problem was encountered which forced slowing the pumping rate. Maximum pressure rise was approximately 400 to 500 PSI as the pill was pumped into the formation.

The well was brought back on stream after 36 hours and produced at the rate of 100 barrels per hour for two days. Flow rate was then increased to approximately 30,000 barrels and has remained there for over six months except for a slow rate decrease that is a function of the reservoir flow properties.

Samples were taken on a regular basis during early flowback and once a day thereafter. Figure 3 shows the I-131 scintillation count data vs. barrels of brine produced. A well defined curve with a maximum at around 1450-1500 barrels of brine flow results.

Samples of the brine were also used for determination of the phosphonate concentrations in solution. These are included in Table 2. In this squeeze treatment there was never the tremendous backflow of the calcium phosphonate that had been observed in the earlier squeeze job. This is probably because the phosphonate pill was pushed far enough back into the reservoir to smear it out over a large volume of the sand grains. After 8 days of production at 30,000 BPD the inhibitor concentration dropped to about 0.15 mg/l and has remained at that level for over six months (Table 2).

The successful application of the phosphonate pill has allowed the Gladys McCall well to be produced at the maximum rate available from the reservoir. Over five million barrels of brine have already been produced using this second pill with apparently no reduction in protection from CaCO₃ deposition.

LABORATORY STUDIES AND THEORETICAL CONSIDERATIONS

In order to prepare for the field work numerous

material from the producing formation at the Gladys McCall well was analyzed by x-ray diffraction and by scanning electron microscopy and microprobe analyses. Results are in Table 3. From the information in Table 3 it can be concluded that the producing formation is sandstone cemented by secondary quartz with a small amount of clay. The porosity was found to be about 20%, similar to Brea sandstone. This core material was used in all laboratory column studies of the Gladys McCall well.

Column Studies

Column studies were conducted using both ground core samples and intact core plugs. Packed columns using ground core material were quick and easy to prepare and allowed numerous variables to be examined.

For the experiments using ground core material 14 g portions of core material were gently crushed with a porcelain mortar and pestle and packed into 10 ml plastic pipets fitted for use as chromatography columns. These columns were saturated with synthetic brine (1 M NaCl and 0.125 M CaCl₂) and then flushed to remove all traces of drilling fluids. These columns had 40% porosity. After preliminary experiments were run to determine appropriate concentration ranges, the following test of the effect of CaCl₂ overflush was performed by injecting the following into a set of columns:

1. 2.0 ml 15% NaCl as a lead spacer
2. 0.5 ml 3% active Gypton T-132 (pH neutral form of Dequest 2000 from Champion Chem. Houston, Tx.)
3. 0.5 ml 15% NaCl as tail spacer
4. 2.0 ml 15% NaCl as overflush

These columns were shut in for 8 hrs at 85°C (185°F) and then slowly backflushed with 40 ml of synthetic brine at 85°C. Columns were then cut into 10 sections and each was analyzed for remaining inhibitor. Next, the 2 ml of 15% NaCl overflush (4. above) was replaced by 2 ml of 15% CaCl₂. All experiments were run in duplicate, at least. The overall mass balance of inhibitor in these experiments was generally 90%, or better.

A fourfold enhanced inhibitor retention is shown in Figure 4 as a consequence of using a CaCl₂ overflush. This corresponded to about 40% of the inhibitor which was loaded. For this reason a CaCl₂ overflush planned to be used in the inhibitor squeeze at the Gladys McCall well. Similar column experiments were conducted using core material from the Deles No. 1 well in the Hitchcock Field. These core materials were calcite cemented. Little difference was observed between the NaCl and CaCl₂ overflush regimes.

Columns of intact Gladys McCall core material were also prepared. Rectangular pieces of core material 1 cm x 1 cm x 8 cm were cut and embedded in Epoxy in a 1 in OD steel pipe. The end cap pieces and reducers on the pipe were all filled with Epoxy and later drilled to allow 1/8" OD high

core material (Figure 5). This system was used to evaluate the adsorption and desorption of inhibitors. Columns were first saturated with synthetic brine (1 M NaCl, 0.125 M CaCl₂, pH = 4.1) and then flushed at 95°C until all traces of drilling fluid were removed. A pill dosage of 0.95 ml of 2% inhibitor in 0.125 M CaCl₂ and 1 M NaCl was injected into the core. The intact core column was shut in for 24 hrs at 95°C.

Desorption of inhibitor into flowing synthetic brine at 95°C was monitored for about 10,000 pore volumes, or sixteen liters. The result is plotted in Figure 6. After 300 pore volumes the inhibitor concentration dropped to about 1 mg/l (as phosphonic acid) and remained approximately constant for another 7000 pore volumes. After about 7000 pore volumes the concentration of inhibitor in the brine rapidly dropped to below detection limit.

Saturation Index

The driving force for CaCO₃ precipitation under any solution conditions can be represented by the saturation index, SI, defined by:

$$SI = \log \frac{(Ca^{2+})(CO_3^{2-})}{K_{sp}} \quad (1)$$

+: brine supersaturated, may form scale
 0: equilibrium
 -: brine subsaturated, scale may dissolve

where (Ca²⁺) and (CO₃²⁻) represent molar concentrations of calcium and carbonate, respectively, and K_{sp} is the conditional solubility product of CaCO₃.¹ The solubility product in Equation 1 is a function of temperature T, pressure P, and ionic strength IS, or total dissolved solids TDS. At the pH values normally encountered in geopressured brines, the ionic calcium concentration in Equation 1 can be replaced by the total calcium, T_{Ca}, which is easily measured. The divalent ionic carbonate concentration, (CO₃²⁻), in Equation 1 can be expressed in terms of bicarbonate (HCO₃) and pH using the second ionization constant of carbonic acid. Below about 8.3 pH, bicarbonate is essentially the same as alkalinity (Alk), which is also readily measured. Finally, by appropriate algebraic substitution the pH can be expressed in terms of Henry's law constant, the alkalinity, and the gas phase partial pressure of carbon dioxide. Odde and Tomson (1982)⁴ have determined least squared curves for the various equilibrium constants as a function of T, P, and TDS. The SI for most gas and oil well brines has been shown to be:¹

$$SI = \log(T_{Ca} Alk^2 / P_{CO_2}) + 5.89 + 1.549 \times 10^{-3} T - 4.26 \times 10^{-5} T^2 - 7.44 \times 10^{-3} P - 2.526 IS^2 + 0.920 IS \quad (2)$$

where T_{Ca} (molar) = (mg/l Ca)/40,000; Alk (molar) = (mg/l HCO₃)/61,000; IS (molar) = conductance (μmho/cm)/66,667 = (mg/l TDS)/58,500; and X_{CO₂} = volume or mole fraction of CO₂ in gas phase, with pressure, P, in psia and temperature, T, in °F.

A similar equation for pH was derived.¹ All of the variables of Equation 2 are readily

measurable. A simple kit to measure Ca, Alk, and TDS on site is available from LaMotte Chemical Co. (Chestertown, Maryland) and the gas phase percent carbon dioxide can be easily measured by e.g. Drager tubes or gas chromatography. The T and P are generally available at the sample point. To facilitate the use of Equation 2 a nomograph was constructed² and a slide rule is available from Shell Canada (Calgary, Alta, Canada). At initial shut in conditions the downhole SI in Equation 2 is theoretically zero for any formation which is calcite cemented. Therefore, a change in saturation index, ASI, was defined as the sum of the changes resulting from P, T, Ca, Alk, PCO₂, and TDS, independently:²

$$ASI = SI_f - SI_i \quad (3a)$$

$$= \Delta SI_P + \Delta SI_T + \Delta SI_{Ca} + \Delta SI_{Alk} + \Delta SI_{CO_2} + \Delta SI_{TDS} \quad (3b)$$

During production in the absence of scale formation the only variables which change from bottom hole to well head are T and P. Nomographs to calculate ASI were also developed.² A semi-quantitative correlation of scale formation process vs SI was developed from field and laboratory studies. For ASI values between zero and 1.1 to 1.4 scale will probably not start to form in equipment free of scale. For ASI values between about 1.4 to 2.3 scale can generally be controlled by trace concentrations of inhibitors. Above ASI of about 2.3 it may not be possible to prevent scale with inhibitors (see below). Below ASI = 0 scale will not form, but corrosion will likely be the prime concern, especially if ASI is less than -1.0.

Inhibitors

Rosenstein in 1935 found that extremely low concentrations of metaphosphates could be used to prevent scale formation.³ Today several classes of compounds are used for this substoichiometric (threshold) scale inhibition, e.g.: 1. inorganic polyphosphates; 2. polyphosphate esters; 3. phosphonates; and 4. low molecular weight polyacrylates; and polymaleates.⁴

Most of these threshold inhibitors are surface active. They probably prevent nucleation of a new scale phase by interacting with the forming nuclei and preventing formation of a stable solid phase.⁵ They prevent further growth of an already existing phase by adsorbing onto active growth sites on the surface and preventing incorporation of more lattice ions.⁶ For brines supersaturated with respect to calcite it has been proposed that the lowest concentration of threshold inhibitor needed would be approximately the molar concentration of divalent carbonate, specifically:⁵

$$2(CO_3^{2-})/[z(In^{2-})] < 1.0 \quad (4)$$

where parenthesis represent molar concentrations and z is the average charge on the inhibitor or inhibitor unit used to express concentration. The summation sign in Equation 4 suggests strict

additivity of mixed inhibitors. In laboratory tests compounds and various combinations of compounds from several chemical classes have been shown to prevent nucleation at concentrations similar to those predicted by Equation 4. Yet, no examples have been found of inhibition of calcite nucleation below the limit suggested by Equation 4.

Also, there is an upper limit at which an inhibitor might work. This upper limit is controlled by the solubility of the inhibitor. Inhibitors of precipitation generally form insoluble salts of one of the lattice ions of the phase being inhibited. All of the classes of inhibitors listed above are anions and form insoluble calcium salts. If the concentration of inhibitor is too large a calcium-inhibitor salt (pseudoscale) precipitates. For some of the field systems investigated by the authors this upper limit of inhibitor concentration has been found to be only a few mg/l or less.

DISCUSSION

The development of a successful squeeze method to inhibit calcium carbonate depositions from produced brines has been tested. This method has been shown effective in sandstone reservoirs where there is no calcite in the cementing material. The key ingredient in a successful application appears to be that the calcium phosphonate needs to be formed in the reservoir at some distance away from the well bore. This can be done by carefully selecting the amounts of preflush to move the brine which contains calcium ion away from the well bore and pumping in excess afterflush to force the phosphonate into the reservoir where it interacts with calcium from the formation water to form the insoluble calcium phosphonate.

There is considerable room for improvement in the development of the inhibitor squeeze technique. For example, we are not sure that the 10% NaCl preflush and afterflush couldn't be done with regularly available oil field brine. Precautions would need to be taken however to see that the Ca and Fe levels in the brine were low. Also, the use of a small pore size filter (preferably 2 microns or less) is a must to prevent blocking of the well bore from any particles in the oil field brine.

The mixing data available from the first successful squeeze job at Gladys McCall is quite interesting. The sodium concentration (Figure 1) rises more sharply than expected and then doesn't appear to remain at the high concentration as long as it should. In fact, the sodium concentration has returned almost to its normal formation brine level by the time 900 barrels of brine have been produced, whereas we'd expect high values to cover at least a five hundred barrel spread. Its apparent peak at the same point as the phosphonate peak also is unexpected.

In the second squeeze inhibitor test the iodine peaks around 1500 barrels (Figure 3) which is about 300 barrels later than would be expected. It is interesting to note however, that if you integrate the area under the iodine curve over 90%

of the iodine has returned by the time 3000 barrel of fluid have been produced.

At flowing wellhead conditions the concentration inhibitor, 0.15 mg/l (1.7×10^{-7} M), is only 15% greater than that predicted by Equation 4.^{1,5} This agreement may be fortuitous, but is similar to the lowest concentration found in laboratory tests.^{1,4} As noted before, with each inhibitor squeeze scale began to form in the low pressure side of the surface equipment at 4 to 5 mos. This occurred as the ASI increased above 2.3 to 2.5 (Equation 3),^{1,2} but an additional 0.25 mg/l of inhibitor was sufficient to prevent scale in the low pressure region of the surface equipment. When greater than 1 mg/l of inhibitor was injected in the surface equipment calcium-inhibitor pseudoscale formed in the filter units.

CONCLUSIONS

1. Phosphonate inhibitor squeeze can be used to successfully prevent formation of CaCO_3 scale in the tubing and surface equipment of brine wells which would normally have scaling problems. Inhibitor concentrations of only 0.15 mg/l have effectively prevented scale formation in the production tubing.
2. Treatments can be successfully carried out in reservoirs where the sand grains are not cemented with calcite.
3. Extreme care should be taken to form the insoluble calcium phosphonate in the reservoir so that it won't block the well bore.
4. The use of a calcium chloride overflush can be avoided by using the natural dispersion properties of the reservoir to induce the mixing of the injected inhibitor with calcium in the formation brine. This greatly simplifies an inhibitor squeeze.

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**TABLE 1-SUMMARY OF BRINE CHEMISTRY OF THE DOE
GEOPRESSURED-GEOTHERMAL GLADYS MCCALL DESIGN WELL
LOCATED IN THE CRAB LAKE FIELD, CAMERON PARISH, LA.**

Parameter (Units)	Value
Temperature Bottom Hole (°F)	298
Temperature Leaving Separator (°F)	264
Pressure Bottom Hole (psi)	12910
Pressure at Disposal Well (psi)	250
Depth (ft)	15600
Alkalinity (mg/l as HCO ₃ ⁻)	547
Calcium (mg/l Ca ²⁺)	4130
Chloride (mg/l Cl ⁻)	57900
Conductivity (μhos/cm)	126,360 @ 25°C
TDS(mg/l)	96340
Hardness (mg/l as CaCO ₃)	12000
Iron (mg/l)	35
Silica (mg/l SiO ₂)	135
Specific Gravity (g/cm ³)	1.062 @ 25° C
Sulfate (mg/l)	ND, < 5 ppm
Sulfide (mg/l)	ND, < 1 ppm
CO ₂ -Gas (Volume %)	7.6
S.I. at Disposal Well	2.55*

*Well will scale heavily if inhibitor is not added, particularly at higher temperatures. Also, when the flow was increased to over 15,000 BPD, the ΔSI at wellhead increased to over 1.2 and scale formed in the production tubing in the absence of inhibitors (see Reference 2 for details).

**TABLE 2-PHOSPHONATE CONCENTRATION IN GLADYS MCCALL
BRINE FLOWBACK AFTER SECOND SUCCESSFUL PILL
TREATMENT**

Brine (B)	Phosphonate (mg/l)
306	NONE
406	NONE
606	200
806	175
1,026	110
1,086	124
1,146	128
1,206	168
1,266	146
1,326	138
1,366	132
1,406	134
1,506	172
4,750	6.4
48,473	0.8
225,632	0.17
628,401	0.13
4,426,428	0.14

**TABLE 3--X-RAY DIFFRACTION (XRD) AND MICROPROBE
ANALYSES OF GLADYS MCCALL CORE SAMPLES**

<u>Mineral</u>	<u>XRD*</u>	<u>Microprobe</u>
Quartz	91%	Present as sand grain and a cement
Calcite	0%	None found
Feldspar	6%	As pure albite ($\text{NaAlSi}_3\text{O}_8$) and pure orthoclase (KAlSi_3O_8)
Kaolinite	2-3%	Pure
Illite		Pure
Chlorite	Trace	Aluminum-rich

*From Geopressured-Geothermal Drilling and Testing Plan, Vol. II, Technadril-Fenix Scisson-Gladys McCall No. 1 Well--U.S. Department of Energy, Nevada Operations Office, Technadril-Fenix & Scisson, January, 1982.

△ NH₄ 1 unit = 100 mg/l

○ Mg 1 unit = 25 mg/l

□ K 1 unit = 200 mg/l

▲ P 1 unit = 50 mg/l

● Na 1 unit = 5000 mg/l

■ Ca 1 unit = 500 mg/l

I-131 TRACER FROM GLADYS McCALL INHIBITOR SQUEEZE

LABORATORY INHIBITOR SQUEEZE CORE COLUMN SYSTEM

DEQUEST 2000 INHIBITOR SQUEEZE

GLADYS McCALL CORE, 15367 ft.

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