CROP CARE

Atlox Rheostrux™ 100 & Atlox Rheostrux 200

Superior rheology modifiers for non-aqueous formulations.

Croda's range of Rheostrux agents offer superior structuring as they result in better stability, rheology profile and suspensibility performance. All this is achieved with ease of use and flexibility.

Oil dispersion formulations (OD) are fast becoming indispensable in the agrochemical industry as a way of formulating hydrolytically unstable actives. They are, however, notoriously difficult to structure effectively due to the high density of the active ingredients. This often leads to an unacceptably high viscosity formulation which still sediments to some degree. Croda's Rheostrux agents offer a solution to this problem, allowing easy formulation of pourable yet stable ODs.

Physical Properties

Product	Melting temperature	Recommended use level	Oil compatibility
Atlox Rheostrux 100	85°C	2-4%	Vegetable oil Paraffinic oil
Atlox Rheostrux 200	83°C	2-4%	Vegetable oil Methylated seed oil (MSO)

Process Advantages

Traditionally non-aqueous formulations are structured using clays and silicas. Atlox Rheostrux 100 & 200 are an excellent alternative due to their ease of use. The main processing advantages over clays and silicas are:

- They are incorporated at the beginning (they are heated with the oil first before other ingredients are added)
- No need to heat the active or other ingredients in formulation
- No issue with dust
- Do not require milling in order to structure therefore long milling times are not necessary*
- Both products shear thin when subjected to high shear meaning they can be processed in the mill but do not degrade

*Only need to mill a formulation containing an Atlox Rheostrux agent if the active ingredient requires a smaller particle size.

Compatibility with Crop Oils

The two Rheostrux agents were incorporated into four neat oils commonly used in crop care. Bentonite clay was also used as a benchmark for comparison. The optimum inclusion level was determined by the highest viscosity obtainable whilst keeping the formulation pourable.

	Atlox Rheostrux 100	Atlox Rheostrux 200	Bentonite Clay (Benchmark)
Vegetable Oil (Sunflower oil)	3%	3%	3%
Paraffinic Oil (SunAG oil)	2%	4%	4%
MSO (Methylated Seed Oil)	Incompatible	3%	Incompatible
Aromatic Hydrocarbon (Solvesso 200ND)	Incompatible	Incompatible	Incompatible

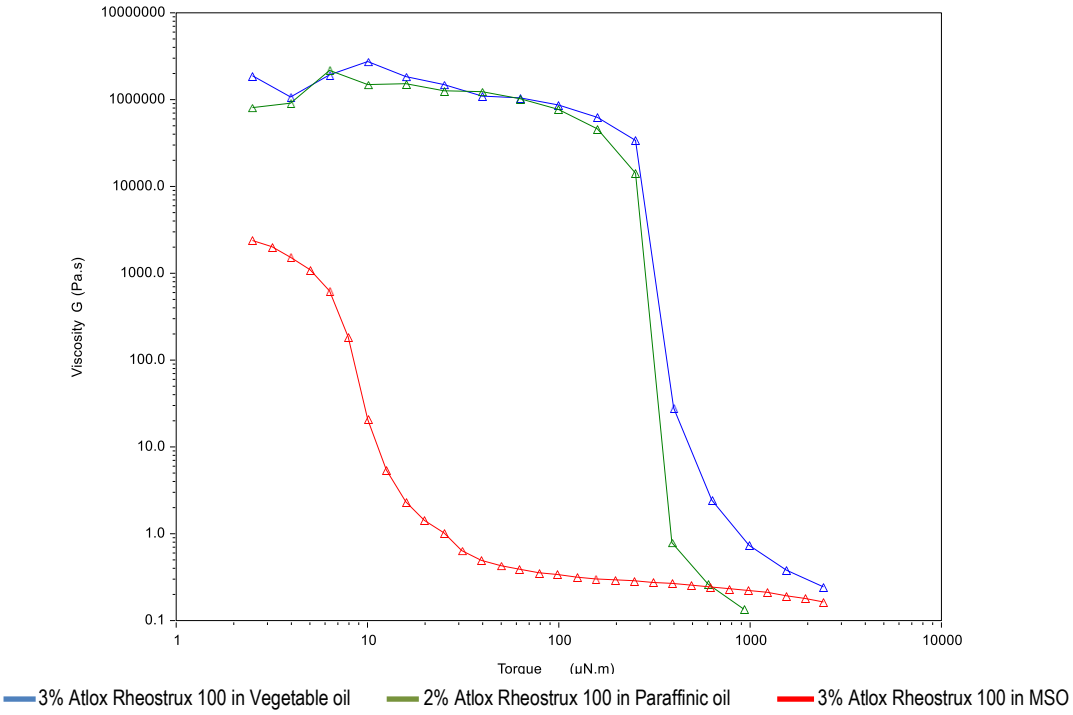
Table 1: Oil compatibility of the Rheostrux agents and recommended inclusion levels compared to a benchmark

From the data shown in table 1, Croda would recommend using either Rheostrux agent for use with vegetable oil. Both products formed acceptable formulations when used with paraffinic oil but Atlox Rheostrux 100 is normally recommended as it is effective at a lower inclusion level. When using MSO, Atlox Rheostrux 200 should be used as this was the only product that was compatible with this oil.

Rheology Modifier and Oil Performance

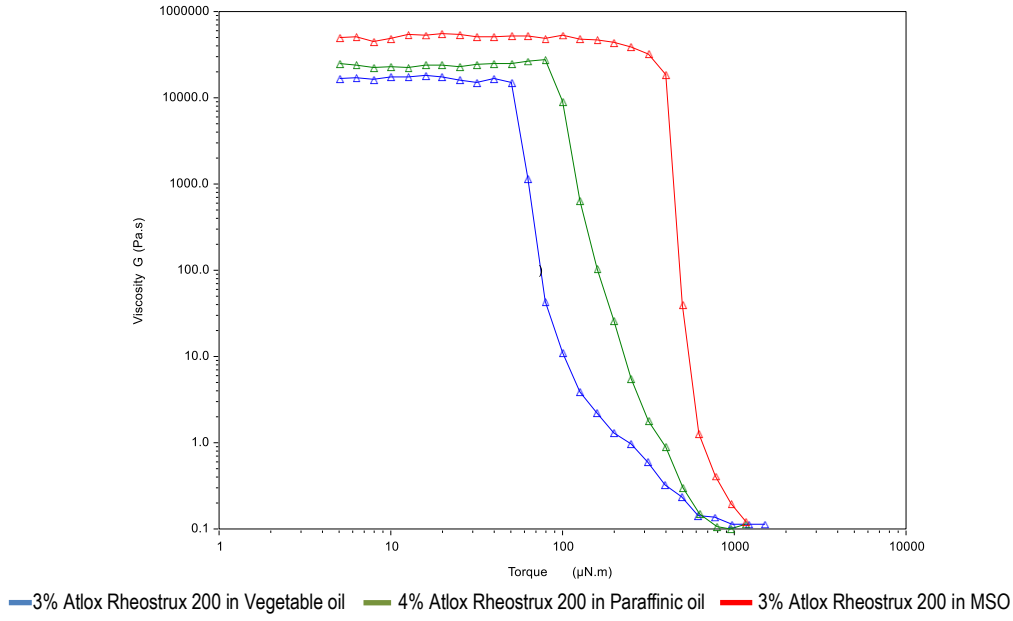
The rheology profiles were looked at using the Rheostrux agents in neat oil. The oils tested were sunflower oil, SunAG oil and MSO.

The tests were conducted on a TA instruments DHR-3 rheometer and their viscosity profile was measured using a flow test. This measured the samples viscosity over a range of torques (force) and was used to look at the storage viscosity and application viscosity of a formulation. As these samples are shear thinning (viscosity decreases with increasing torque), the range over which they shear thin can give insight into how homogeneous a sample's structure is.



Graph 1: Rheology profile using Atlox Rheostrux 100 as the rheology modifier in various crop oils.

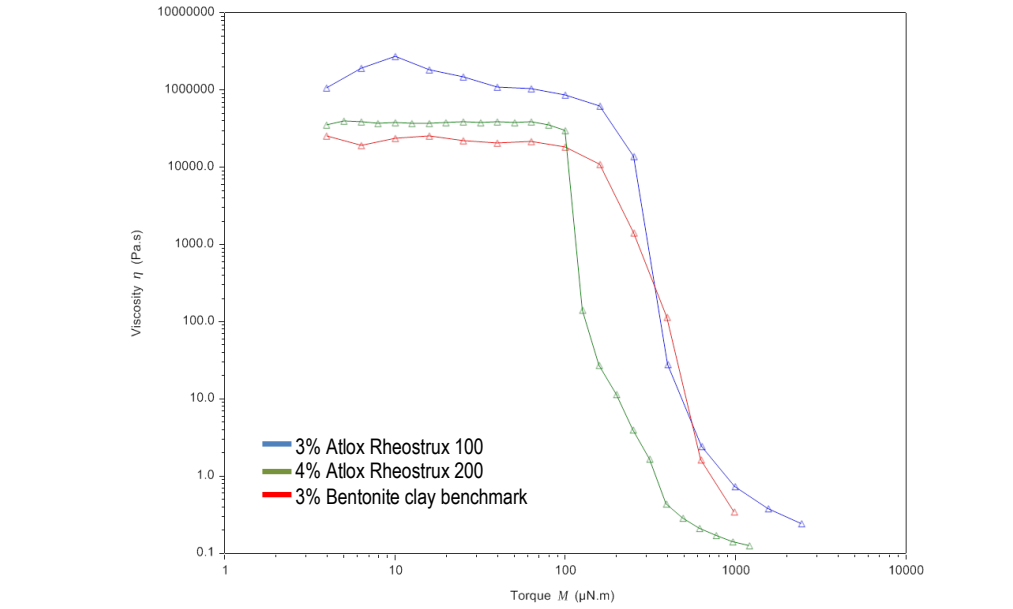
Graph 1 shows that Atlox Rheostrux 100 has good rheology profiles for both sunflower oil (vegetable oil) and SunAG oil (paraffinic oil). Both have a high viscosity at low shear (low torque) which means better stability when the sample is under storage. They both shear thin at high shear meaning they become flowable and can be poured easily during application. Additionally, they shear thin over a narrow range meaning they are homogeneous. Croda recommends using Atlox Rheostrux 100 for vegetable and paraffinic oil but not for MSO.



Graph 2: Rheology profile using Atlox Rheostrux 200 as the rheology modifier in various crop oils.

A good rheology profile for all three oils was achieved when using Atlox Rheostrux 200. All have high viscosity at low shear (low torque) which mean better stability when the sample is stood. They all shear thin at high shear meaning they become flowable and can be poured easily. Additionally, they shear thin over a narrow range meaning they are homogeneous. Croda recommends using Atlox Rheostrux 200 with MSO and vegetable oil, as shown by the excellent rheology profile above. A good rheology profile can also be achieved in paraffinic oil but Atlox Rheostrux 100 is normally recommended instead as it is effective at a lower inclusion level (2% compared to 4%).

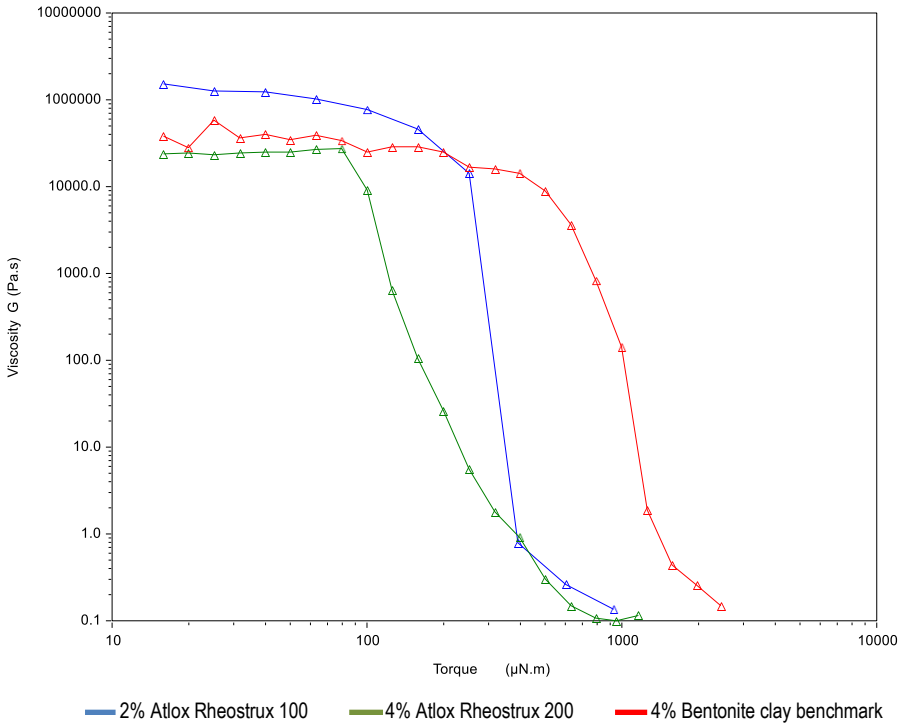
Comparison to the Benchmark (Bentonite clay)



Graph 3: Rheology profile of Atlox Rheostrux 100 and Atlox Rheostrux 200 in **sunflower oil** compared to the clay benchmark

The flexibility of the Rheostrux agents is another advantage over clay. If storage stability is an issue then the level of Rheostrux agent can be increased. Graph 3 shows that Atlox Rheostrux 200 had been increased to 4%. This gives the advantage of a better storage viscosity (leading to better storage stability) while still remaining flowable in the high shear region. However, this cannot be done with clay as increasing it above 3% results in an increase in storage viscosity but the product becomes unflowable in the high shear region and can therefore not be used.

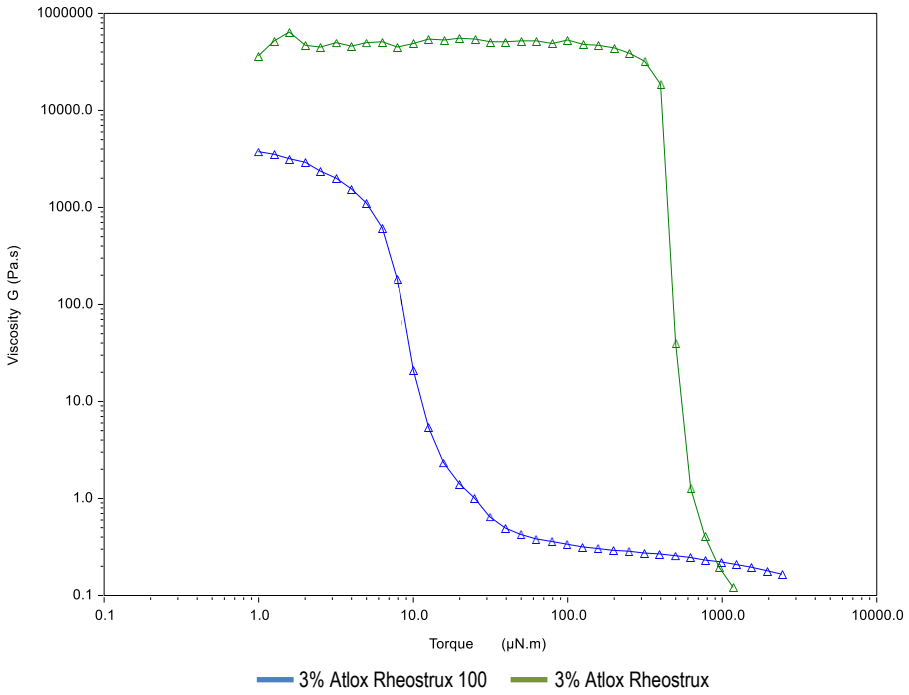
Graph 3 also shows that Atlox Rheostrux 100 has the most desirable rheology profile in sunflower oil. In other vegetable oils this may not be the case so Croda recommends screening both Rheostrux agents in vegetable oils.



Graph 4: Rheology profile of Atlox Rheostrux 100 and Atlox Rheostrux 200 in **SunAG oil** compared to the clay benchmark

Graph 4 shows a good rheology profile can be achieved with Atlox Rheostrux 100 and Atlox Rheostrux 200 in paraffinic oil but Atlox Rheostrux 100 is normally recommended as it is effective at a lower inclusion level (2% compared to 4%).

Graph 4 also shows that the Rheostux agents have a better rheology profile compared to clay in SunAG oil. They all have similar storage viscosities but the clay does not shear thin until a high torque is applied (i.e. beyond the procesing shear region). This results in the clay being harder to pump and therefore harder to use. The level of the clay cannot be reduced as although it will become pourable, sedimentation will start to occur as the storage visocity will also drop.



Graph 5: Rheology profile of Atlox Rheostrux 100 and Atlox Rheostrux 200 in **MSO** compared to the clay benchmark

The rheology profiles shown in graph 5 highlight that Atlox Rheostrux 200 should be used for MSO. As clay could not form a stable product it could not be tested, showing that clay cannot be used in combination with MSO.

Formulation Performance

Both Rheostrux agents were tested in a formulation and compared to a benchmark formulation containing clay as the structurant. Copper oxychloride was used as the active ingredient as this is a high density active and was used at a high inclusion level. This tested the performance of the rheology modifier in harsh conditions.

Formulation containing Atlox Rheostrux 100:

Ingredient	**Function**	**% Inclusion**
Sunflower oil	Continuous phase	43.58
Copper oxychloride	Active ingredient	43.6
Atlas™ G-1086	Oil emulsifier	7.2
Zephrym™ PD2206	Non-aqueous dispersant	0.81
Atlox Metasperse™ 550S	Aqueous dispersant	0.81
Atlox Rheostrux 100	Rheology modifier	4.0

Formulation containing Atlox Rheostrux 200:

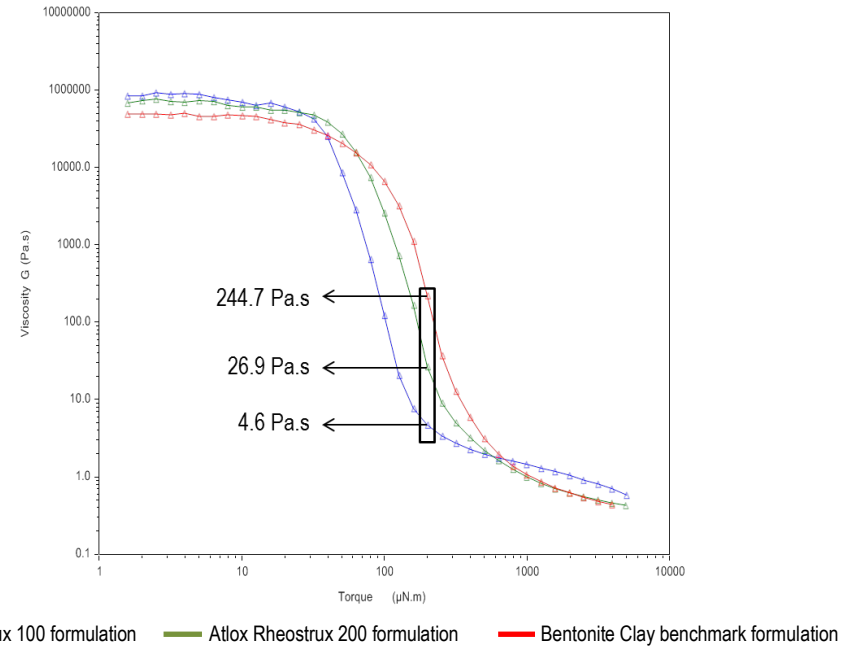
Ingredient	**Function**	**% Inclusion**
Sunflower oil	Continuous phase	44.08
Copper oxychloride	Active ingredient	43.6
Atlas G-1086	Oil emulsifier	7.2
Zephrym PD2206	Non-aqueous dispersant	0.81
Metasperse 550S	Aqueous dispersant	0.81
Atlox Rheostrux 200	Rheology modifier	3.5

Benchmark formulation containing clay:

Ingredient	Function	% Inclusion
Sunflower oil	Continuous phase	44.58
Copper oxychloride	Active ingredient	43.6
Atlas G-1086	Oil emulsifier	7.2
Zephrym PD2206	Non-aqueous dispersant	0.81
Atlox Metasperse 550S	Aqueous dispersant	0.81
Clay benchmark	Rheology modifier	3.0

Rheology Profiles

The formulations were tested using the same method and instrument as the testing done on the rheology modifier in neat oil.



Graph 6: Rheology profiles of the two Atlox Rheostrux agents in formulations compared to the benchmark clay formulation

Graph 6 shows that in the high shear region (above 100 µN.m) both the Croda structurants have shear thinned to a better viscosity than the clay formulation.

Torque of 200 (highlighted on graph 6)
RheoStrux 100 viscosity 4.6 Pa.s
RheoStrux 200 viscosity 26.9 Pa.s
Bentonite clay viscosity 224.7 Pa.s

This means the Atlox Rheostrux products are easily milled/processed and allows a lower torque to be used in the mill meaning less energy is used. The graph also shows that both Croda products have higher rest viscosity. This means that the formulations will be more stable on storage with better suspensibility but due to their shear thinning properties have the advantage of also being pourable systems.

Stability Performance
The stability of all the formulations was assessed after 24 hours, 7 days and 14 days at room temperature (RT), 40°C and 54°C. The stability of all the formulations was assessed according to CIPAC MT 46.1.3. A pass is <20% separation after 14 days at 54°C.

Key

- Stable indicated by ST
- Traces of separation (<1%) oil or water where Tt indicates traces of top separation and Tb indicates traces of bottom separation.
- Separation (>1 and <20%) oil or water where %Sb indicates separation at bottom and %St indicates separation at top
- Broken indicated by Br, where there is complete phase separation or S>20%

Label	24 hours			7 days			14 days		
	RT	40 °C	54 °C	RT	40 °C	54 °C	RT	40 °C	54 °C
Atlox Rheostrux 100 Formulation	ST	ST	ST	Tt	2.5% St	5% St	1% St	4% St	9% St
Atlox Rheostrux 200 Formulation	ST	ST	ST	Tt	4% St	8%St	2% St	6% St	12% St
Benchmark Formulation (Bentonite clay)	ST	ST	Tt	Tt	8% St	13% St	2% St	11% St	Br

Table 2: Stability performance of the two Croda rheology modifiers in formulations compared to the benchmark

Table 2 shows that the formulation containing the Rheostrux agents are more stable than the benchmark formulation containing clay. Both Rheostrux agents pass the accelerated heating test whereas the clay formulation failed.

Suspensibility Performance
Due to OD formulations containing both solid and liquid components both creaming and sedimentation instability was measured in the suspensibility testing. This was done using the CIPAC MT180 method where instability was measured at both the top and bottom of the Crowe tube. The pass criterion is a maximum of 5ml of creaming at the top of the Crowe tube and a maximum of 2ml of sedimentation at the bottom.

	30 minutes	2 hours
Atlox Rheostrux 100 Formulation	1 ml cream 0.5 ml sediment	1.5 ml cream 1 ml sediment
Atlox Rheostrux 200 Formulation	0.5 ml cream 0.5 ml sediment	0.5 ml cream 0.5 ml sediment
Benchmark Formulation (Bentonite clay)	5ml cream 1 ml sediment	5ml cream 1.5 ml sediment

Table 3: Suspensibility performance of the two Rheostrux agents compared to the benchmark formulation containing clay

Table 3 shows the suspensibility results for the three formulations produced. Both Rheostrux agents performed better than the clay benchmark as less creaming and sedimentation was observed with the Croda products.

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Formulating Guidelines

Directions for use

- Heat the Atlox Rheostrux product in the oil ~85°C until a homogeneous liquid is formed
- Leave to cool to allow the product to set hard forming a viscous paste/gel
- Add the remaining ingredients, dispersants, active ingredients etc (no need to heat the active ingredient)
- Stir (1500 rpm) until a homogenous product is formed
- Bead mill the formulation for 15 minutes (3500 rpm)
- Remove from the mill and allow to restructure before testing

Other Considerations

- Appearance of Atlox Rheostrux 100 is a brown powder
- Appearance of Atlox Rheostrux 200 is a slight yellow clear pastel
- Melting temperature can be oil dependant
- Oil and rheology modifier premix must be left to cool to fully structure before milling
- Final formulation requires structuring overnight before storage testing

Atlox Rheostrux 100
Brown powder

Atlox Rheostrux 200
Slight yellow clear pastel

Guideline Formulations

Ingredient	Function	g/L	% w/w
Copper oxychloride	Active ingredient	633.20	43.60
Sunflower oil	Continuous phase	632.90	43.58
Atlas™ G-1086[1]	Oil emulsifier	104.56	7.20
Zephrym™ PD2206[1]	Non-aqueous dispersant	11.78	0.81
Atlox Metasperse™ 550S[1]	Aqueous dispersant	11.78	0.81
Atlox Rheostrux 100[1]	Rheology modifier	58.09	4.00

Supplier: **1. Croda**

Ingredient	Function	g/L	% w/w
Copper oxychloride	Active ingredient	633.30	43.60
Sunflower oil	Continuous phase	632.90	44.08
Atlas G-1086[1]	Oil emulsifier	104.56	7.2
Zephrym PD2206[1]	Non-aqueous dispersant	11.76	0.81
Metasperse 550S[1]	Aqueous dispersant	11.76	0.81
Atlox Rheostrux 200[1]	Rheology modifier	52.50	3.5

Supplier: **1. Croda**

Ingredient	Function	g/L	% w/w
Zinc oxide	Active ingredient	700.00	44.40
MSO	Continuous phase	588.10	37.50
Atlas G1086[1]	Oil emulsifier	115.00	7.30
Atlox 4915[1]	Aqueous dispersant	70.00	4.40
Atlox LP1[1]	Non-aqueous dispersant	70.00	4.40
Atlox Rheostrux 200[1]	Rheology modifier	32.00	2.00

Supplier: **1. Croda**

Summary
Atlox Rheostrux 100 and Atlox Rheostrux 200 are superior alternatives to clays and silicas in oil dispersion formulations offering better stability, rheology and suspensibility performance as well as ease of use.

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