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Technical data sheet

DIPLAST® TM 4

Version: March 2015

Special plasticizer based on Polynt SpA Trimellitic Anhydride

Chemical composition
Tri-n-butyl trimellitate

CAS number
1726-23-4

EINECS number
217-038-0

Specifications

Characteristics	Unit	Value	Test method	
Density at 20°C	g/ml	1.060 - 1.064	GM 012	ASTM D 4052-96
Refractive index n^{20}_{D}		1.490 - 1.494	GM 020	ASTM D 1045-95
Colour	Pt –Co	80 max.	PL02F	ASTM D 1045-95
Acidity	mg KOH/g	0.1 max.	PL02C	ASTM D 1045-95
Water content	%	0.1 max.	GM 010	ASTM E 203-96
Ester content	%	99.5 min.	PL10C	G.C.

DIPLAST® TM 4 is a pale yellow liquid, anhydrous, clear and free from matter in suspension. It is miscible with common organic solvents, practically insoluble in water and miscible and compatible with most of the monomeric plasticizers usually utilised to soften PVC.

The product **DIPLAST® TM 4** due to its nature does not have a shelf life. However it can be stored in appropriate containers at a temperature of approximately 25°C and the exclusion of humidity for at least 1 year, without loosing its chemical properties.

Characteristics and applications of trimellitates

PVC plasticizers based on Polynt SpA Trimellitic Anhydride (Trimellitate plasticizers), offer to users and compounders an optimum performance profile.
Trimellitate plasticizers show an unique balance of properties that can be summarized as follows:

- **Processability and efficiency**
  Comparable with those of many Phthalate plasticizers and better than most Polyester plasticizers.

- **Excellent permanence at high temperatures and retention of mechanical properties.**

- **Low temperature flexibility**
  Unlike Polyester plasticizers, Trimellitate plasticizers provide good flexibility at low temperatures.

- **Permanence and compatibility**
  Trimellitates are more permanent in soft PVC articles than many other plasticizers. They have great compatibility with PVC and have a resistance to extraction in various solvents higher than phthalates.
  Also their migration resistance in PVC in contact with various polymers is higher than that of phthalates.

DIPLAST® TM 4 is a fast gelling plasticizer for PVC. Its outstanding compatibility with PVC allows to satisfy particular requirements (materials with high plasticizer content, reduced processing time, high transparency of the finished articles).
Due to the above mentioned properties, **DIPLAST® TM 4** is particularly suitable to produce plastisols for PVC flooring, where fast gelation is required.

General properties in PVC compounds

The properties of **DIPLAST® TM 4** were evaluated in comparison with those of **DIPLAST® B** (DIBP) and **DIPLAST® NS** (DINP), in PVC formulation containing 50 phr of plasticizer:
All of the specimens were prepared by calendering PVC sheets, which were successively moulded to obtain the thickness required for the different test methods.

Formulation	PVC K70	Plasticizer	Ca/Zn	Stearic Acid
phr	100	50	1.2	0.3

Results

	Test method	DIPLAST® TM4	DIPLAST® B	DIPLAST® NS
Shore "A" hardness (15")	ISO 868	75	77	80
Cold flex °C (Clash & Berg)	ISO/R 458	-12	-10	-26
Extraction resistance				
% weight loss (48h at 70°C)	ISO 175			
• Water		-0.2	-1.3	-0.1
• Aqueous soap 1%		-5.4	-5.3	-0.7
• Olive oil		-4.8	-5.8	-6.8
• Mineral oil		-4.4	-5.4	-5.5
• n-Hexane (24 hours at 23°C)		-5.2	-5.6	-27.6
Volatility (7days at 100°C)	ISO 176	-7.5	-22.5	-6.1
Solution Temperature °C (*)	DIN 53408	93	90	129
Rheological properties				
• Dryblending time 83°C (Mixer P-600 : 100 Rpm)	Brabender Plasticorder	1'34"	1'20''	3'42"
• Gel time (88°C) (Mixer W-50 : 40 Rpm, 48gr)	Brabender Plasticorder	2'28"	2'54"	9'04"
• Fusion Temperature (°C) (Mixer W-50, 5°C/min, 40Rpm)	Brabender Plasticorder	111	109	117

(*) Solution temperature determined with dispersion of resin: two grams of PVC are placed in 48 grams of plasticizer and the solution is heated at 1°C/min.

Rheological tests

The data report below were obtained by using a *TA Instruments AR 1500 Reometer*:

- Flow measurements
- Oscillatory tests

<u>PVC Plastisols formulation:</u>

Emulsion PVC	100
Plasticizer	70

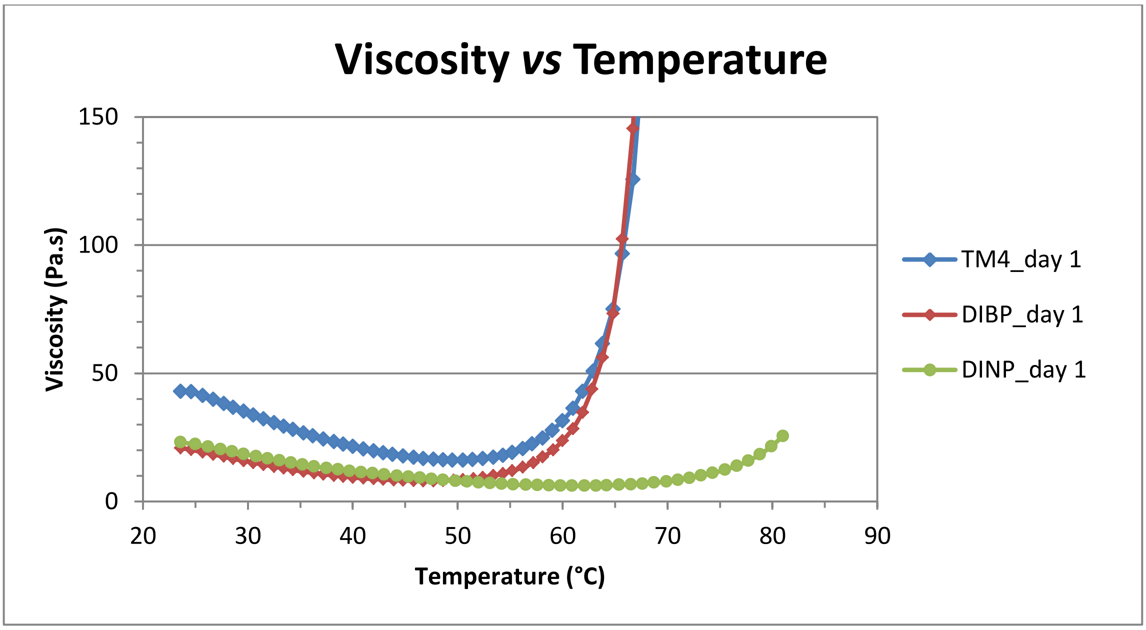
Flow measurements with temperature ramp (ramp flow temperature, T: 23-80 ° C)

The rheological properties of the **DIPLAST® TM 4** have been evaluated in comparison with those of **DIPLAST® B** (DIBP) and the **DIPLAST® NS** (DINP).

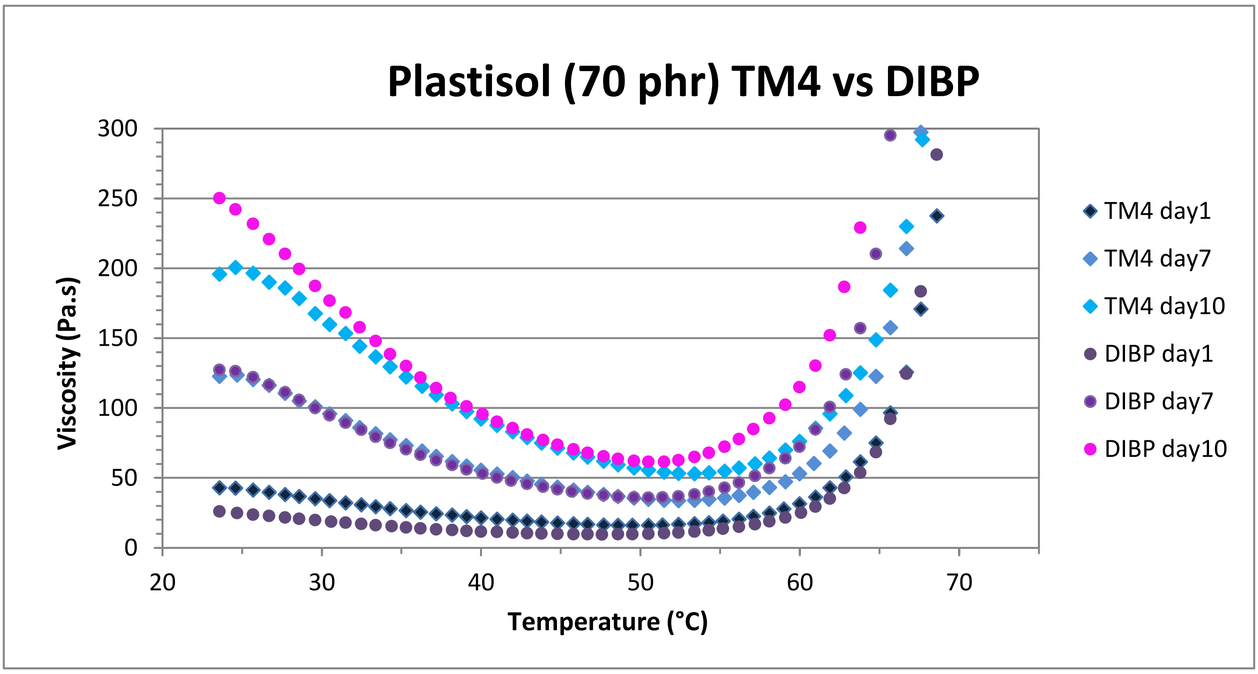
Flow measurements conditions

1) Conditioning step T: 23°C for 2 min
2) Temperature ramp step T: 23 – 80°C
3) Temperature gradient of 2°C/min
4) Ramp duration 10 min
5) Shear stress (Pa) 10
6) Sampling rate 10 s

The graph below shows the viscosity *vs* temperature curve for different PVC plastisols, where the plasticizer's content is 70 phr. The highly compatible plasticizers **TM4** and **DIBP** impart a sharp viscosity increase starting from 50°C, while the DINP plastisols viscosity increases smoothly from 65°C onward. These data demonstrate that **DIPLAST® TM 4** is as fast gelling as **DIBP,** and much faster than **DINP**.



According to the graph below, the rheological properties of the **DIPLAST® TM 4** have been evaluated in comparison with those of **DIPLAST® B** (DIBP).
The graph shows the viscosity *vs* temperature curve for different plastisols (plasticizer's content 70 phr) over time, after 1 hour, 1 day and 7 days of aging in thermostatic chamber (T: 23 ° C, relative humidity 50%).



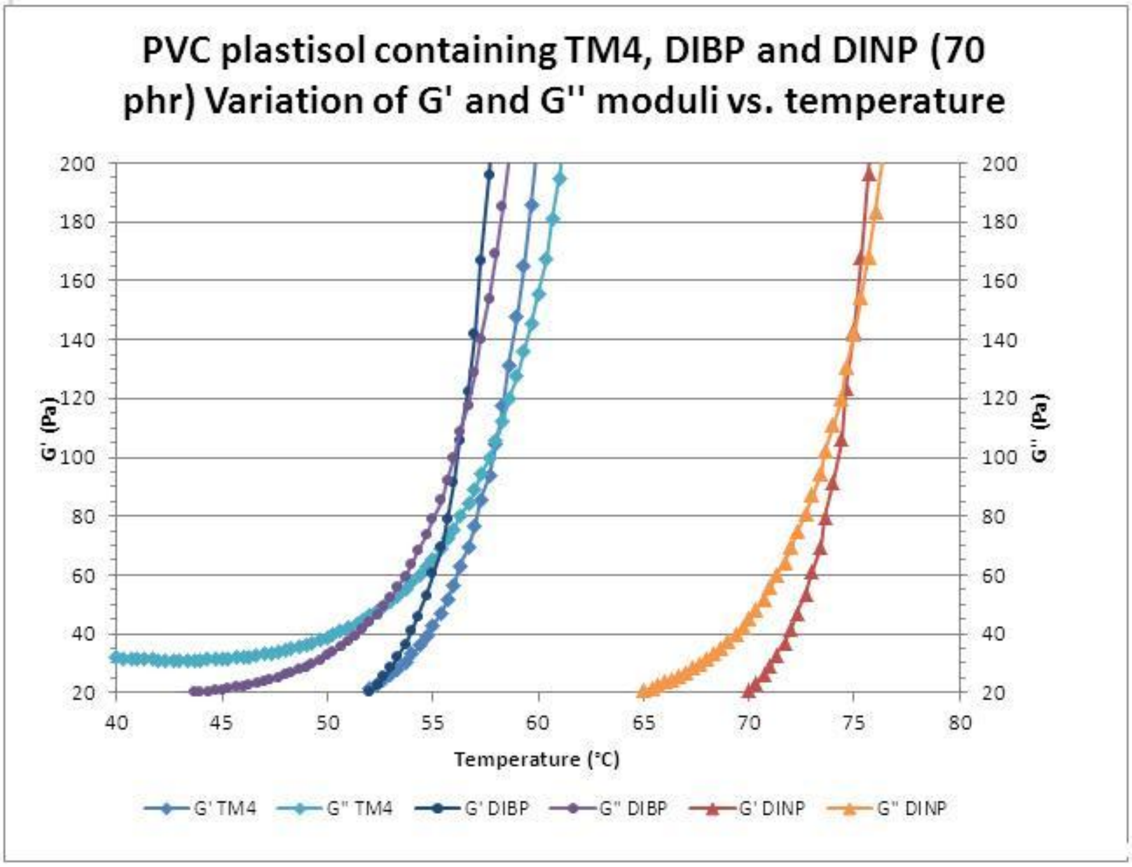
The figure shows an increase of viscosity of the plastisol over time according to the high compatibility of the plasticizer with the PVC (fast fusing plasticizers).

Oscillatory tests with temperature ramp (temperature sweep T: 23-90 °C, 2°C/min)

Oscillatory rheological experiments at different temperatures and over a wide range of frequencies have been used to investigate the gelation process and, more particularly, the sol-gel transition of various PVC plastisols.
The viscoelastic behavior of PVC plastisols containing **DIPLAST® TM4, DIBP** and **DINP** has been evaluated by oscillatory tests, where the variation of G' (storage or elastic modulus) and G'' (loss modulus) as a function of temperature was measured.
Being G' and G", respectively, the elastic and the viscous component of a material, the gel point can be defined as the time when G' and G" cross over.

Oscillatory time sweeps

1) Conditioning step T: 23°C for 2 min
2) temperature ramp step T: 23-90 °C
3) Ramp rate 2°C/min
4) sampling rate 10 s
5) Oscillation stress (Pa) 10
6) Frequency (Hz) 1,0



The temperatures of gelation (gel points) are reported in the table below; these data confirm the ones obtained by the above reported flow tests, i.e. **DIPLAST® TM4** is as fast gelling as DIBP, and much faster than **DINP**.

Formulation (100 phr PVC; 70 phr plasticizer)	**Oscillatory tests Temperatures of gelation (gel point) (°C)**
DIPLAST® B (DIBP)	57
DIPLAST® TM4	62
DIPLAST® NS (DINP)	79

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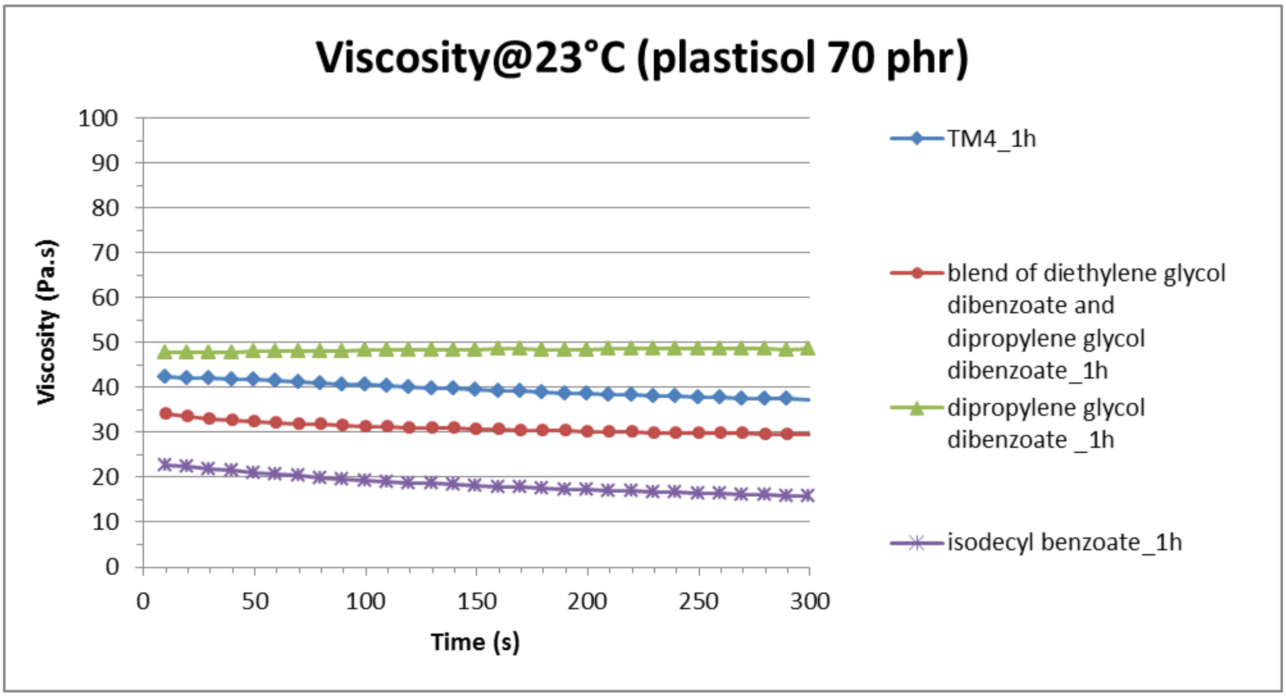
Comparison DIPLAST® TM4 with Benzoates

The rheological properties of the **DIPLAST® TM 4** have been evaluated in comparison to those of the benzoates, the results are reported in the following table:

Formulation [1]	Viscosity 23°C [2] (mPas)			Oscillatory tests [3] Gel point (°C)	Flow Test [4] Shear rate max. (°C)
	1 hours	1 day	7 days		
DIPLAST® TM4	40.000	39.000	46.000	62	56
blend of diethylene glycol dibenzoate and dipropylene glycol dibenzoate	36.000	32.000	26.000	60	54
dipropylene glycol dibenzoate	48.000	47.000	44.000	55	44
isodecyl benzoate	20.000	8.000	5.000	70	63

[1] 100 phr PVC emulsion; 70 phr plasticizer.
[2] Viscosity of the plastisol over time, after 1 hour, 1 day and 7 days of aging in thermostatic chamber (T: 23°C, relative humidity 50%).
[3] Oscillatory tests; temperature oscillation sweep; T: 23-90°C; 2°C / min, it was reported the temperature value recorded in time when G '(storage modulus or elastic modulus) and G''(dissipative modulus or loss modulus) cross over (**gel point**).
[4] Flow Measurement; flow temperature ramp; T: 23-80°C; Time: 10 min, it was reported the temperature recorded at the time when it reaches the maximum value of shear rate (minimum viscosity of the plastisol).

The viscosities at 23°C of the products are compared after 1 hour of maintenance in the thermostated chamber (T: 23°C, relative humidity 50%) and the data are shown in the graph below:



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Comparison of plastisol Diplast TM4 at different concentrations.

The rheological properties of the plastisol **DIPLAST® TM4** were evaluated at different concentrations (phr) of plasticizer and the results are described below.

Formulation [1]	Viscosity 23°C [2] (mPas)			Oscillatory tests [3] Gel point (°C)	Flow Test [4] Shear rate max. (°C)
	1 hours	1 day	7 days		
DIPLAST® TM4 65phr	54.000	54.000	90.000	57	52
DIPLAST® TM4 70phr	40.000	39.000	46.000	62	56
DIPLAST® TM4 80phr	16.000	16.000	18.000	68	58

[1] 100 phr PVC emulsion; X phr plasticizer.
[2] Viscosity of the plastisol over time, after 1 hour, 1 day and 7 days of aging in thermostatic chamber (T: 23°C, relative humidity 50%).
[3] Oscillatory tests; temperature oscillation sweep; T: 23-90°C; 2°C / min, it was reported the temperature value recorded in time when G '(storage modulus or elastic modulus) and G''(dissipative modulus or loss modulus) cross over (**gel point**).
[4] Flow Measurement; flow temperature ramp; T: 23-80°C; Time: 10 min, it was reported the temperature recorded at the time when it reaches the maximum value of shear rate (minimum viscosity of the plastisol).