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Analysis of Nucleating Agent (NA-27) in Polypropylene Samples By ICP MS

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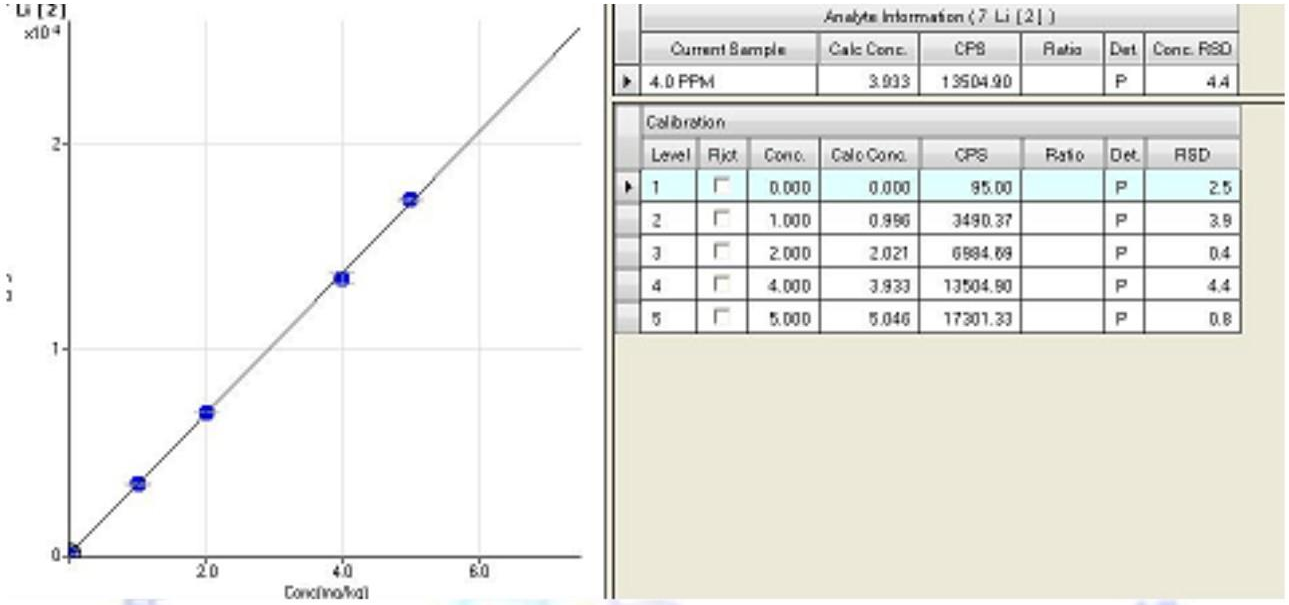
Abstract

Analysis of NA27 additive in Polypropylene samples having I-168 additive NA-27 is an advanced nucleating agent for polypropylene, providing superior mechanical properties, easier dispersion and reduced interaction with metal stearate. It is effective even in talc-filled polypropylene. Nucleating agents/Clarifiers NA 27 added in polypropylene grades (1110MAS /3120MA/ 4080MA/3650MN/3400MN) to give high degree of crystallinity resulting in increased mechanical properties such as hardness, elasticity modulus etc, and improve optical properties such as transparency. In many grades where I-168 and NA-27 are added together, it is challenging to quantify the I-168 and NA-27 additives separately. As both of the additives are phosphorus based and do not separate out in HPLC or WDXRF. To overcome this issue a new method has been developed for the analysis of NA27 contents in polypropylene samples having I-168 additives.

Keywords- HPLC, WXRF, NA-27 additives etc.

Experimental work:

ICP-MS 7700 series is used to determine the Li content in NA-27 additive. A calibration curve was prepared for **Li** content ranging 0.0 to 5 mg/Kg concentration.



Analyte Information (7 Li [2])

Current Sample	Calc Conc.	CPS	Ratio	Det.	Conc. RSD
4.0 PPM	3.933	13504.90		P	4.4

Calibration

Level	Rjct.	Conc.	Calc Conc.	CPS	Ratio	Det.	RSD
1		0.000	0.000	95.00		P	2.5
2		1.000	0.996	3490.37		P	3.9
3		2.000	2.021	6984.69		P	0.4
4		4.000	3.933	13504.90		P	4.4
5		5.000	5.046	17301.33		P	0.8

Calibration curve of Li content

After calibration about 100 gm Polypropylene resin sample was weighted nearest 0.01 grams in a crucible and kept in muffle furnace at 600 ^{o}C for 1 hour and residue is digested with concentrated nitric acid and diluted with DM water as per desired analysis range. Digested sample was analyzed for lithium content over ICMPS operating at following conditions:

RF power	1550W
Sample depth	8 mm
Carrier gas flow	1.12 Lit/min
Nebulizer pump	0.1 rps
Rinse speed	0.50 rps
Rinse time	30 sec
Uptake time	45 sec
Uptake speed	0.50 rpm

Different standard of NA 27 were prepared (By mixing powder, NA27, I-168 weight by weight followed by single extrusion over cast line) and analyzed for Li content and respectively NA 27 concentration.

S No	Sample details	Li Concentration (mg/Kg)	Concentration NA-27 (mg/Kg)	Deviation From standard Value	% Deviation from standard
1	Pure NA 27	1.100 %			
2	PP with 0 ppm NA-27	0	0		
3	PP with 100 ppm NA-27	1.102	100	0	0.00
4	PP with 200 ppm NA-27	2.115	192	8	3.86
5	PP with 250 ppm NA-27	2.65	241	9	3.64
6	PP with 300 ppm NA-27	3.49	317	-17	-5.76
7	PP with 0350ppm NA-27	3.99	363	-13	-3.64

Repeatability of NA-27analysis in PP sample found as below:

S. No	Sample ID	Li (mg/Kg)	NA 27 (mg/Kg)
1	PP -II 879	1.75	159
2	PP -II 879	1.94	176
3	PP -II 879	2.11	192
4	PP -II 879	1.88	171
5	PP -II 879	1.96	178

Uncertainity of NA-27 measurement (at the level of 175 mg/Kg) is ± 11 mg/kg (@95% confidence level).

Conclusion-

Newly developed method is accurate and reliable for analysis of NA-27 additives. NA 27 contains two Phosphate ester compound and Organic compound. Out of two phosphate ester components of NA27, one component is around 30-40% and named as 12H- Dibenzo [d,g][1,2,3] diox aphosphocin, 2,4,8,10-tetrakis(1,1dimethylethyl)-6-hydroxy-, 6-oxide, lithium salt having a molecular formula $C_{29}H_{42}LiO_4P$ and molecular weight 492.556142 [g/mol].Nucleating agent is added along with other additives like anti oxidants I-1010, I-168 and emulsifier GMS-90. Blends of phenolic and phosphorus based antioxidants (Like I-1010 and I-168) are analyzed by HPLC and WD XRF techniques.

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