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Production of 60 kg ADN and 20 kg GAP using current methods

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Summary

The aim of this part of the project was to produce the required amount of ADN and GAP and to deliver it to respective partner in August 2011.

EuB has produced 60 kg of crude ADN using the current standard production method.

28 kg neat ADN was delivered to FOI 17/06/2011 and 9 kg of neat ADN and 3 kg of prilled ADN was delivered to ICT 24/08/2011. 5 kg of neat ADN and 1 kg of prilled ADN will be sent to TNO during September 2011 for their work with crystallisation and/or prilling.

The remaining quantity of the produced ADN will be crystallised or prilled using the method chosen later on in the project and then sent to the participants.

EuF has produced 20 kg of GAP using the current standard production method. 6 kg will be sent to FOI and 2 kg to TNO in the last part of August. The remaining 12 kg will be sent to ICT and AVIO (6 kg each) when they need it for their work in the project.

The tables below summarises the status of ADN and GAP deliveries respectively.

Status of EuB delivery of neat ADN.

| Partner | Neat ADN needed (kg) | Delivery status |
|---------|----------------------|--------------------------------|
| FOI | 18 + 10* | Delivered 17/06/2011 |
| ICT | 9 | Delivered 24/08/2011 |
| TNO | 5 | Will be delivered in September |

* All ADN to be prilled by FOI will first be sent to FOI and then to respective partner. FOI will use 18 kg for their own development work in the project.

Status of EuF delivery of GAP.

| Partner | GAP needed (kg) | Delivery status |
|---------|-----------------|-------------------------|
| FOI | 6 kg | August 2011 |
| ICT | 6 kg | Not yet ordered by ICT |
| AVIO | 6 kg | Not yet ordered by AVIO |
| TNO | 2 kg | August 2011 |

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1 Introduction

The estimated quantities of ADN and GAP needed for the HISP project are 60 kg of ADN and 20 kg of GAP.

FOI will prill ADN and send it, via EuB, to interested partners. FOI, TNO and ICT will work with ADN particle production and will thus need neat ADN. AVIO will only be using FOI prilled ADN.

The required amounts of ADN and GAP have been produced by EuB and EuF respectively, using their current production methods. The materials will be transported to the appropriate project partners, in time for each partner to fulfil their part of the project. The first deliveries will be made during the second half of August 2011.

2 Current production methods

2.1 ADN production method

The current standard production method for ADN at EuB follows the route shown in the figure below.

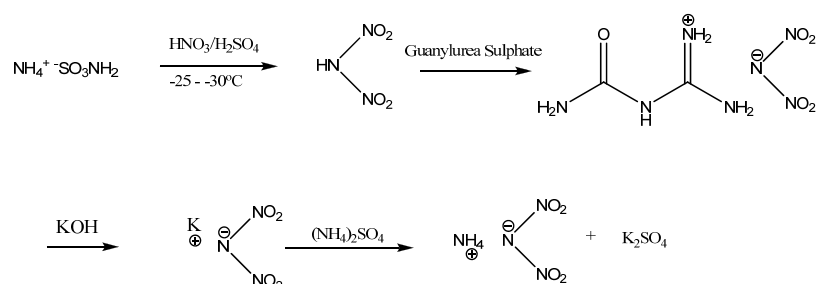


Figure 1. Synthesis of ADN.

The ADN produced by this method should fulfil the following specifications:

Table 1. Characterization of ADN.

| Characterization | Unit | Specification | Results for lot 20119036 |
|---------------------|------|-----------------------------|------------------------------------|
| ADN content | % | Min. 98 | 100 |
| Moisture (50°C) | % | Max. 0.4 | 0.1 |
| Potassium content | % | Max 0.4 | 0.2 |
| Appearance | | White to yellowish crystals | White to weakly yellowish crystals |
| Melting point (DSC) | °C | Min 92 | 92 |

The ADN used for the HISP project will be distributed as follows:

Table 2. Required quantities of ADN.

| Project partners | ADN quantity | Order number, date |
|------------------|--|--|
| FOI | 18 kg neat | May 30 th |
| ICT | Totally 18 kg First delivery: 9 kg neat, 3 kg prilled by FOI | S4306, June 7 th |
| TNO | Totally 6 kg 5 kg neat and 1 kg prilled by FOI | S4305, June 4 th |
| AVIO | 18 kg prilled | Not yet received. Will be delivered in November according to plan. |

2.2 GAP production method

The current standard production method for GAP at EuF follows the route shown in the figure below.

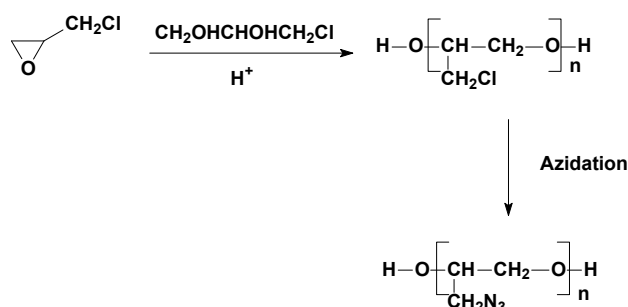


Figure 2. Synthesis of GAP.

The GAP produced by this method should fulfil the following specifications:

Table 3. Characterization of GAP.

| Characterization | Unit | Specification | Results for lot 14S10 |
|---------------------------------|--------------------|---------------------|-----------------------|
| Aspect | | Clear yellow liquid | Conform |
| Gardner colour | | ≤ 5 | 5 |
| Mw | | 1700-2300 | 1909 |
| Average weight molecular weight | | | |
| Mn | | 1400-2000 | 1685 |
| Average number molecular weight | | | |
| Q = Mw/Mn | | ≤ 1.3 | 1.1 |
| Polydispersity | | | |
| OH content | kg/OH | 1000/1400 | 1160 |
| Water content | % | ≤ 0.05 | 0.04 |
| Volatile matter | % | ≤ 0.1 | ≤ 0.05 |
| Chlorine content | % | ≤ 0.2 | 0.0 |
| Vacuum test (193 h. / 100°C) | cm ³ /g | ≤ 3.0 | 0.7 |

The GAP used for the HISP project will be distributed as follows:

Table 4. Required quantities of GAP.

| Project partners | GAP quantity | Order number, date |
|------------------|--------------|--------------------------------|
| FOI | 6 kg | 621061, April 19 th |
| ICT | 6 kg | Not yet received |
| AVIO | 6 kg | Not yet received |
| TNO | 2 kg | 41004430, July 1 st |

3 Production of ADN (EuB)

3.1 Production of 60 kg ADN

60 kg of ADN was manufactured using the current standard production method at EuB. The production was carried out in pilot scale and it was completed June 9th. The batch, lot 20119036, was analysed according to the specifications shown in Table 1. The results from the analysis are also shown in Table 1.

3.2 Transportation of ADN

28 kg of neat ADN was sent to FOI at June 17th. Of those kilos, 4 kg were prilled for the needs of ICT and TNO and sent back to EuB. The prilled ADN arrived at EuB on August 11.

In order to keep transportation costs down, neat and prilled ADN will be sent together with the same transport. 13 kg (9 kg neat and 3 kg prilled product) was sent to ICT 24/08/2011, and 6 kg (5 kg neat and 1 kg prilled product) will be sent to TNO in September 2011. The remaining quantity of ADN (prilled or neat) will be delivered in November 2011 according to plan.

4 Production of GAP (EuF)

4.1 Production of 20 kg GAP

GAP has been manufactured at industrial scale at EuF, according to the current standard production methods (Figure 2), that is with a two step reaction where the first step is synthesis of PECH (polymerization of epichlorohydrin) and the second one is the azidation of PECH.

The produced lot (14S10) has been analysed and the product fulfils the specification as shown in Table 3.

4.2 Transportation of GAP

The total required amount of GAP for all the project partners is 20 kg. It will be distributed to the partners as shown in Table 4.

The GAP is ready to be delivered. So far, two orders have been received in EuF (from FOI and TNO). The product will be sent to them, according to what has been agreed on in the order confirmation. AVIO and ICT have still not told when they want their product, but it will be sent to them in time for them to fulfil their parts in the project.

5 Conclusions

The production of ADN and GAP needed for this project have been successfully completed. The products fulfil the specifications and will be sent to the partners, in time for them to fulfil their tasks.