# Synthesis and Characterization of Some New 1,3,4-Oxadiazole and 1,2,4-Triazole Derivatives Based on 3,4,5,6 Tetrachlorophthalimide

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**Abstract:**
Several new derivatives of 1,3,4-oxadiazoles and 1,2,4-triazoles linked to 3,4,5,6-tetrachlorophthalimide moiety were synthesized through following multisteps. The first step involved preparation of 3,4,5,6-tetrachlorophthalimide via reaction of 3,4,5,6-tetrachlorophthalic anhydride with urea at high temperature. Treatment of the resulted imide with ethyl chloroacetate in the second step afforded tetrachlorophthalimidyl ester which inturn was introduced in reaction with hydrazine hydrate in the third step, producing the corresponding acetohydrazide. The synthesized acetohydrazide was introduced in different synthetic paths including treatment with phenyl isothiocyanate or reaction with carbon disulfide in alkaline solution then with hydrazine hydrate to afford the new 1,2,4-thiazoles, while introducing of acetohydrazide in reaction with carbon disulfide in alkaline solution under reflux or in reaction with benzaldehyde producing a new Schiff base. Treatment of the new Schiff base with acetic anhydride afforded the new 1,3,4-oxadiazoles.
**Keywords:** synthesis, imide, 1,3,4 – Oxadiazole, 1,2,4 - Triazole

# تحضير وتشخيص بعض من مشتقات 4,3,1 - اوكسادايازول و 4,2,1- ترايازول الجديدة اعتماداً على المركب 6,5,4,3 – رباعي كلوروفثال ايماید

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**الخلاصة:**
تم في هذا البحث تحضير عدد من مركبات4,3,1 – اوكسادايازول ومركبات 4,2,1 – ترايازول جديدة المرتبطة بمكونة 6,5,4,3 – رباعي كلوروفثال ايماید. تم تحضير هذه المركبات باتباع عدة خطوات حيث تضمنت الخطوة الاولى تحضير المركب 6,5,4,3 – رباعي كلوروفثال ايماید من خلال تفاعل 6,5,4,3 – رباعي كلوروانهدريدالفثاليك مع اليوريا في درجات حرارة عالية. اما في الخطوة الثانية فقد تم معاملة الايماید المحضر مع كلوروخلات الاثيل لانتاج الاستر خلات رباعي كلورو فثال ايميديل وهذا بدوره تمت معاملته في الخطوة الثالثة مع الهيدرازين المائي لانتاج مركب الاسيتوهايدرازيد المقابل. تم ادخال مركب الاسيتوهايدرازيد المحضر في عدة مسارات تحضيرية حيث ان تفاعلاته مع كل من فنيل ايزوثايوسيانات ومع ثاني كبريتيد الكاربون في الوسط القاعدي ثم مع الهيدرازين المائي اسفرت عن تكوين مشتقات 4,2,1 – ترايازول الجديدة بينما اسفرت تفاعلاته مع كل من البنزالديهايد تكون قاعدة شيف. ان معاملة قاعدة شيف الجديدة مع انهدريد الخليك اسفر عن تكوين مشتقات 4,3,1 –اوكسادايازول الجديدة.

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## 1. Introduction

The synthesis of high nitrogen containing heterocyclic systems has been attracting increasing interest over the past decade because of their utility in various applications such as propellants, explosives, pyrotechnics and especially chemotherapy [1]. Among these heterocycles the 1,3,4-oxadiazole motif is of particular value in materials science, agrochemistry and in pharmaceutical chemistry [2]. A number of synthetic routes have been developed for 1,3,4-oxadiazole [3,4]. Majority of these are based upon cyclodehydration of diacylhydrazines [5]. Similarly 1,2,4-triazoles and their fused heterocyclic derivatives has received considerable attention owing to their synthetic [6,7] and effective biological importance [8] thus, a large number of 1,2,4-triazole containing ring system have been used in a wide variety of applications as inhibitors of corrosion, polymers [9,10], drugs candidates and as synthetic dyes.

In addition, phthalimides are bicyclic nitrogen heterocycles constitute an important class of compounds with a variety of applications and a wide range of properties. Generally they are used as starting materials and intermediates for synthesis of many types of alkaloids and pharmacophores, in polymers [11], synthesis of pesticides and lately are being under intense biomedical research due to their important biological effects [12].

In view of above mentioned facts and in continuation of our work on the synthesis of new cyclic imides linked to different heterocycles, it was planned in this work to incorporate the (1,3,4-oxadiazole or 1,2,4-triazole) ring system into tetrachlorophthalimide ring starting from tetrachlorophthalimide which introduced in reaction with ethyl chloroacetate producing tetrachlorophthalimidyl acetate ester. The later was converted to a useful intermediate having hydrazide function by treatment with hydrazine hydrate, which inturn was readily introduced in different synthetic paths affording the desired new heterocyclic derivatives.

## 2. Experimental

### 2.1. Instruments

FTIR spectra were performed on a Shimadzu FTIR 8400 Fourier Transform Infrared spectrophotometer. $^{1}$HNMR and $^{13}$CNMR spectra were recorded on a Bruker, Ultrashield 300 MHz spectrometer. Melting points were determined on Gallenkamp melting apparatus and were uncorrected.

### 2.2. Chemicals

All chemicals were of analytical reagent grade and were used without further purification.

#### 2.2.1. Preparation of 3, 4, 5, 6-tetra chloro phthalimide (1)

3,4,5,6-Tetrachlorophthalic anhydride (0.02 mol, 5.72 g) and urea (0.05 mol, 3g) were mixed, homogenized in a flask, and heated in an oil bath [13]. The mixture began melting at (230-240) $^{o}$C then was slowly brought to 260$^{o}$C until it completely turned into liquid. After this region, a mass of yellow crystals appeared and swelled to as 3-4 times than its initial volume. The obtained solid was dispersed in (100 mL) of water, filtered, and finally recrystallized from water.

#### 2.2.2. Preparation of 3, 4, 5, 6-tetra chloro phthalimide potassium salt (2)

Compound (2) was prepared by dissolving (0.01mol, 2.85g) of compound (1) in (5 mL) of DMF. The clear solution was added to alcoholic potassium hydroxide solution with stirring. The obtained white precipitate was filtered and dried, then recrystallized from acetone.

#### 2.2.3. Synthesis of ethyl-2-(N-(3, 4, 5, 6-tetra chlorophthalimidyl)) acetate (3)

Ethyl chloroacetate (0.01 mol, 2.5 mL) was added to a mixture of (0.01 mol, 1 g) of imide potassium salt (2) dissolved in (10 mL) of dimethyl sulfoxide. The mixture was thoroughly mixed then was heated under reflux for one hour [14]. After cooling, (10 mL) of water was added and the obtained product was filtered, dried then recrystallized from ethanol.

#### 2.2.4.Synthesis of 2-(N-(3, 4, 5, 6-tetra chloro phthalimidyl)) acetohydrazide (4)

A solution of the prepared ester (3) (0.01mol, 3.6 g) in ethanol (50 mL) was mixed with hydrazine hydrate (0.02 mol, 98%) [14]. The reaction mixture was refluxed for ten hours then added to ice-water and the product was filtered, dried then recrystallized from methanol.

**2.2.5. Synthesis of 2-(N-(3, 4, 5, 6-tetra chloro phthalimidyl)) 1,3,4-oxadiazole-5-thiol (5)**

The acetohydrazide (4) (0.002 mol, 0.72 g) was dissolved in a solution of potassium hydroxide (0.22g) in ethanol. (20 mL) of carbon disulfide (2 mL) was added with stirring [14]. The reaction mixture was refluxed for 20 hours then the solid residue was treated with water followed by filtration. The filtrate was neutralized by dilute HCl and the formed precipitate was filtered, dried, and finally recrystallized from benzene.

**2.2.6. Synthesis of 2-(N-(3, 4, 5, 6 - tetra chloro phthalimidyl ) ) N' – benzylidine - aceto hydrazide (6)**

A mixture of (0.001 mol, 0.36 g) of acetohydrazide (4) and (0.001 mol, 4 mL) of benzaldehyde in absolute ethanol containing two drops of glacial acetic acid was refluxed for three hours with stirring [15]. The solvent was evaporated and the precipitate formed was washed with ether, dried, and recrystallized from ethanol.

**2.2.7. Synthesis of 2-phenyl 3-acetyl-5-(N-3, 4, 5, 6-tetrachlorophthalimidyl) methylene - 2,3-dihydro-1,3,4-oxadiazole (7)**

A mixture of Schiff base (6) (0.003 mol) and acetic anhydride (10 mL) was heated under reflux for four hours. After the reaction mixture attained room temperature, excess acetic anhydride was decomposed by adding water and the mixture was stirred for further 30 min. The separated product was filtered, washed with water, dried, and recrystallized from ethanol.

**2.2.8. Synthesis of 4-amino-5-N-(3, 4, 5, 6-tetra chlorophthalimidyl) methylene-2, 3-dihydro -1, 2, 4-triazole-3-thiol (8)**

To a solution of potassium hydroxide in absolute ethanol, acetohydrazide (4) (0.003 mol, 1 g) and carbon disulphide (0.006 mol, 0.45 mL) were added and the mixture was agitated for 12 hrs. [14]. The resulted precipitate was filtered, washed with ether and dried under vacuum and used in the next stage. A suspension of the prepared potassium salt, hydrazine hydrate (1.5 mL) and water (1 mL) was heated under reflux for five hours. The resulted solution was diluted with (25 mL)of water followed by acidification with acetic acid producing a white precipitate which was filtered, washed with water, dried, and recrystallized from dimethyl formamide (DMF).

**2.2.9. Synthesis of 4- N-benzylidine -5- (N-(3, 4, 5, 6-tetrachloro phthalimidyl) methylene- 2,4-dihydro-1,2,4-triazole-3-thiol (9)**

A mixture of compound (8) (10mmol, 4.13g) in absolute ethanol (25 mL) with (10 mmol, 1mL) benzaldehyde and few drops of glacial acetic acid was refluxed for four hours then the resulted mixture was poured into ice-water under stirring [14]. The obtained precipitate was filtered off and washed with water, dried, and finally recrystallized from ethanol.

**2.2.10. Synthesis of 5-(N-(3, 4, 5, 6-tetra chloro phthalimidyl)) methylene-4-phenyl-2,4-dihydro-3H-1,2,4-triazole-3-thione (10)**

A mixture of acetohydrazide (4) (10 mmol, 4.13 g) and excess of phenyl isothiocyanate (15 mmol, 1.8 mL) in ethanol (25 mL) was refluxed for four hours [14]. The resulting solution was cooled to room temperature and the formed precipitate was filtered, dried, and recrystallized from ethanol. Physical properties of compounds (5-10) are listed in Table -1-.

**3. Results and Discussion:**

The present work is directed towards synthesis of new heterocyclic derivatives through incorporation of 1,3,4-oxadiazole and 1,2,4-triazole cycles into 3,4,5,6-tetrachlorophthaimide ring.

Performing this target was achieved through following multi step synthesis which its steps are outlined in Scheme (1). The first step involved the synthesis of unsubstituted 3,4,5,6-tetrachlorophthalimide via reaction between 3,4,5,6-tetrachlorophthalic anhydride and excess of urea at high temperature (230-240)$^{o}$C then raised to 260$^{o}$C.

The structure of the yellow crystals of compound (1) was assigned on the basis of FTIR, $^{1}$HNMR, and $^{13}$CNMR spectral data. The FTIR spectrum showed characteristic band at 3217 cm$^{-1}$ belongs to ν(N-H) imide and this is very important proof for success of imide formation. Other bands appeared at 1774 cm$^{-1}$, 1708 cm$^{-1}$, and 1658 cm$^{-1}$ which belongs to asym. ν(C=O) imide, sym. ν(C=O) imide, and ν(C=C) aromatic respectively [16]. $^{1}$HNMR spectrum of compound (1) showed signals at δ=(11.1-11.9)ppm belong to (N-H) while

$^{13}$CNMR spectrum showed signals at δ=(128.3-150) ppm and (165) ppm belong to aromatic carbons and (C=O) imide respectively.

In this work, compound (1) represents the main important key compound from which all the newly synthesized compounds are prepared. Thus compound (3) is the first compound that derived from compound (1). Synthesis of compound (3) involved two steps; in the first one compound (1) was converted to its potassium salt (2) via treatment with alcoholic potassium hydroxide. The resulted salt (2) was introduced in reaction with ethyl chloroacetate under reflux condition in the second step producing the desired ester (3).

Conversion of compound (1) to its potassium salt increased the nucleophilicity of nitrogen atom during nucleophilic attack of the salt on alpha carbon in ethyl chloroacetate in the second step which represents the conversion of the unsubstituted imide (1) to N-substituted imide (3), firstly and secondly, the N-substituted side chain (-$CH_2CO_2Et$) contains the good leaving group (-OEt) which we work on in the next step.

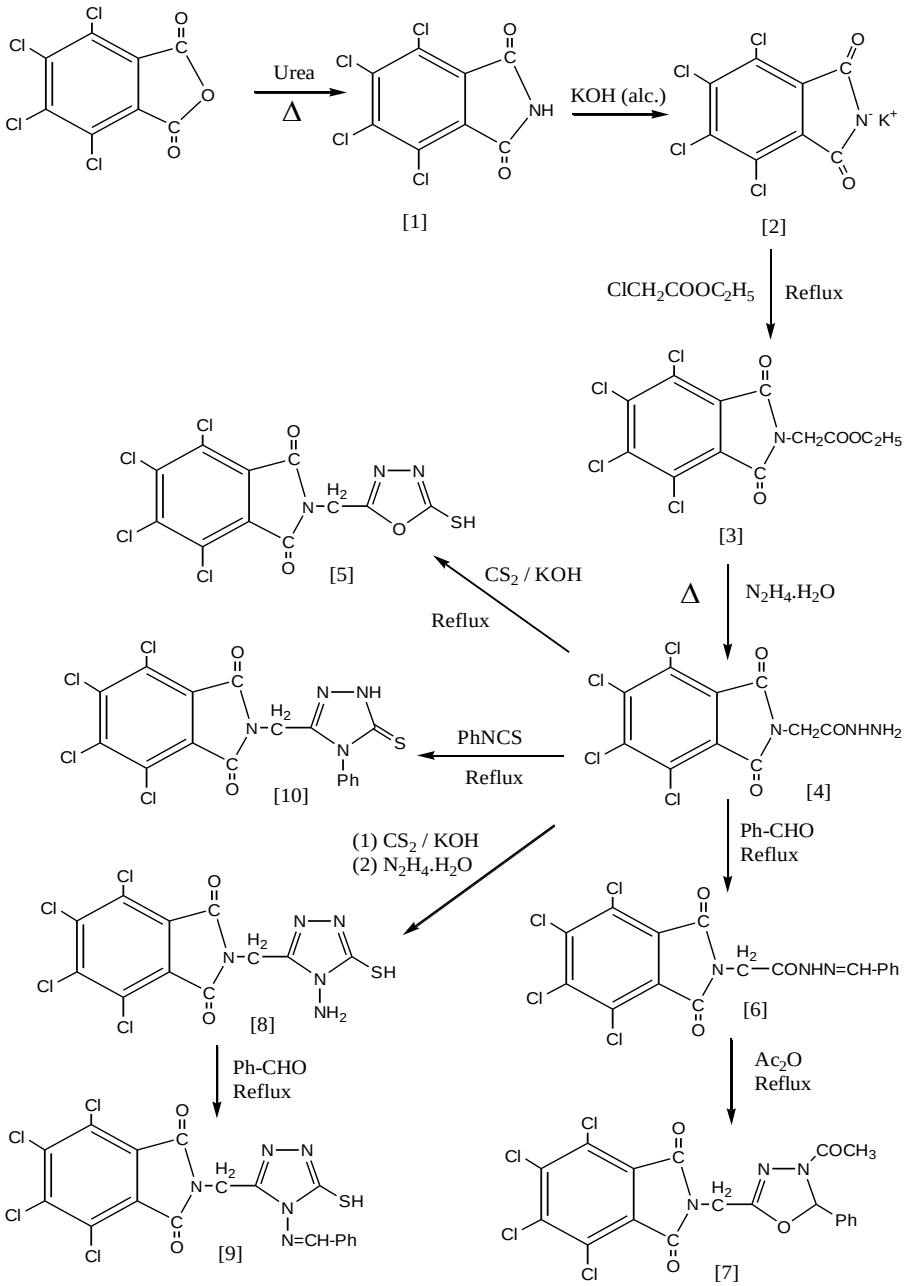
FTIR spectrum of compound (3) showed disappearance of band at 3217 $cm^{-1}$ belongs to ν(N-H) and appearance of characteristic absorption bands at 1747 $cm^{-1}$, 1246 $cm^{-1}$, and 1122 $cm^{-1}$ which belong to ν(C=O) ester, asym. ν(C-O) ester, and sym. ν(C-O) ester respectively. These two points are excellent proofs for the success of ester compound (3) formation. Other bands appeared at 1770, 1720, and 1581 $cm^{-1}$ belong to asym. and sym. ν(C=O) imide and ν(C=C) aromatic [16,17].

The third step in this work involved conversion of the prepared ester (3) to the corresponding acetohydrazide (4), by treatment of compound (3) with hydrazine hydrate in absolute ethanol under reflux condition. The reaction represents nucleophilic substitution reaction in which the nucleophile ethoxy group is displaced by the stronger nucleophile (-$NHNH_2$).

FTIR spectrum of compound (4) showed disappearance of absorption bands belong to both ν(C=O) and ν(C-O) ester and instead appearance of two characteristic absorption bands at 3394 $cm^{-1}$ and 3275 $cm^{-1}$ belong to ν(NH-$NH_2$). These two points are very important proofs for the success of compound (4) acetohydrazide formation. Other absorption bands appeared at 1774, 1720, 1658, 1643, and 1307 $cm^{-1}$which belong to asym. ν(C=O) imide, sym. ν(C=O) imide, ν(C=O) amide, ν(C=C) aromatic and ν(C-N) imide respectively [17].

The next step in this work involved introducing of compound (4) in different synthetic paths producing various new heterocyclic (1,3,4-oxadiazole and 1,2,4-triazole) all of them contain 3,4,5,6-tetrachlorophthalimide moiety.

The first synthetic path involved introducing of compound [4] in reaction with carbon disulfide in alcoholic potassium hydroxide solution under reflux [14] producing compound (5) which contain oxadiazole ring linked to 3,4,5,6-tetrachlorophthalimide component through methylene group.




**Scheme 1-** Preparation of the new desired compounds.

FTIR spectrum of compound (5) showed absorption band at 3414 $cm^{-1}$ belongs to (NH) formed due to tautomerism, other bands appeared at 1770, 1705, 1616, 1550, 1168, and 613 $cm^{-1}$ which belong to asym. ν(C=O) imide, sym. ν(C=O) imide, ν(C=N), ν(C=C) aromatic, ν(C-O-C) and ν(C-S) respectively.

$^{1}$HNMR spectrum of compound (5) showed signals at δ=1.68 ppm and 3.1 ppm belong to NH proton and $CH_2$ protons respectively while $^{13}$CNMR spectrum showed signals at δ= 41, (111-129) belong to ($CH_2$) and aromatic carbons. Signals belong to (C=N), (C=S), and (C=O) appeared at (159.7-184.2) ppm.

The other synthetic path in the present work involved introducing of compound (4) in reaction with benzaldehyde in absolute ethanol with the presence of few drops of the catalyst glacial acetic acid under reflux producing the new Schiff base (6).

FTIR spectrum of compound (6) showed absorption bands at 3417, 1782, 1728, 1681, 1620, and 1573 $cm^{-1}$which due to ν(N-H) amide, asym. ν(C=O) imide, sym. ν(C=O) imide, ν(C=O) amide, ν(C=N), and ν(C=C) aromatic respectively [15].

$^{1}$HNMR spectrum of compound (6) showed signals at δ=4.8 ppm which belong to ($CH_2$)

protons, signals at δ=(7.5-8.8) ppm belong to aromatic protons and NH proton, and signal at δ=8.7 ppm belongs to imine proton.

It is known that Schiff bases can be introduced successfully in reactions with many reagents through active imine group, thus the prepared new Schiff base (6) introduced in reaction with acetic anhydride under reflux producing compound (7). The reaction here involved nucleophilic attack by imine nitrogen in Schiff base on carbonyl group in acetic anhydride followed by nucleophilic attack of amide carbonyl group through oxygen atom on the positive carbon leading to ring closure and finally producing compound (7).

FTIR spectrum of compound (7) showed many absorption bands at 1762, 1724, and 1708 $cm^{-1}$ due to asym. ν(C=O) imide, sym. ν(C=O) imide, and ν(C=O) amide respectively. Other bands appeared at 1620, 1573, 1338, 1211, and 1153 $cm^{-1}$ due to ν(C=N), ν(C=C) aromatic, ν(C-N) imide, and both asym. and sym. ν(C-O-C) respectively [16].

$^{1}$HNMR spectrum of compound (7) showed signals at δ=2.7 ppm belong to ($CH_3$) protons, signals at δ=4.2 ppm belong to ($CH_2$) protons and the proton in oxadiazole ring, finally signals at δ=(7.8-8.2) ppm belong to aromatic protons. $^{13}$CNMR spectrum showed signals at (22.48, 64, and 82) ppm belong to $CH_3$, $CH_2$, and carbon in oxadiazole ring. Other signals appeared at (125.9-134) ppm and 161 ppm belong to aromatic carbons and (C=N), (C=O).

The third synthetic path in this work involved introducing of compound (4) in reaction with carbon disulfide and alcoholic potassium hydroxide producing potassium dithiocarbazinate salt which in turn was treated with hydrazine hydrate under reflux condition producing the target compound (8).

FTIR spectrum of compound (8) showed bands at 3468 $cm^{-1}$ and 3414 $cm^{-1}$ belong to ($NH_2$), bands at 1782, 1712, 1635, and 1620 $cm^{-1}$ due to asym. ν(C=O) imide, sym. ν(C=O) imide, ν(C=N), and ν(C=C) aromatic.

$^{1}$HNMR spectrum of compound (8) showed signals at δ=1.25 ppm belong to $NH_2$ proton, signal at δ=4.1 ppm belong to ($CH_2$) protons, and signal at δ=5.2 ppm belong to NH and SH protons [17].

Compound (8) represents a new heterocyclic compound contains 1,2,4-triazole ring linked to phthalimide moiety through methylene group and also containing amino group which was introduced in the next step reaction..

The fourth synthetic path involved introducing of compound (8) in reaction with benzaldehyde in absolute ethanol producing compound (9) which is a new heterocyclic compound containing three active moieties tetrachlorophthalimide, 1,2,4-triazole ring, and Schiff base moiety. Synthesis of compound (9) involved a nucleophilic attack of amino group in compound (8) on carbonyl group in benzaldehyde followed by elimination of water molecule forming imine bond and producing the new Schiff base (9).

FTIR spectrum of compound (9) showed absorption bands at 1778 $cm^{-1}$ and 1728 $cm^{-1}$ due to asym. ν(C=O) imide and sym. ν(C=O) imide, bands at 1681, 1573, and 1369 $cm^{-1}$ due to ν(C=N), ν(C=C) aromatic, and ν(C-N) imide.

The fifth synthetic path in this work involved introducing of compound [4] in reaction with phenyl isothiocyanate producing compound (10) which is a new heterocyclic compound contains 1.2.4-triazole ring and tetrachlorophthalimide moiety linked together through methylene group. The synthesis was proceed through nucleophilic attack of hydrazide amino group on electron - deficient carbon in phenyl isothiocyanate followed by second nucleophilic attack causing ring closure which subsequently leading to produce compound (10).

FTIR spectrum of compound (10) showed absorption bands at 3464, 1732, 1631, 1597, and 1330 $cm^{-1}$ which belong to ν(N-H), ν(C=O) imide, ν(C=N), ν(C=C)aromatic, and ν(C-N) imide respectively. $^{1}$HNMR spectrum of compound (10) showed signal at δ=4.5 ppm belong to ($CH_2$) protons, signals at δ=(6.9-7.6) ppm belong to aromatic protons, and signals at δ=9.8 and 11 ppm belong to NH and SH protons.

$^{13}$CNMR spectrum of compound (10) showed signals at (65.7-67) ppm belong to ($CH_2$), signals at (116-138) ppm belong to aromatic carbons, and signals at 141, 155, and 187 ppm belong to (C=S), (C=N), and (C=O) imide respectively. Physical properties of the prepared compounds (1-10) are listed in Table -1-.

**Table 1-** Physical properties of compounds (1-10).

| Crystallization solvent | Yield % | m.p. $^{o}$C | Colour | Comp. No. |
|---|---|---|---|---|
| Water | 85 | >300 | Yellow | 1 |
| Acetone | 80 | - | White | 2 |
| Ethanol | 70 | 208-210 | Yellowish white | 3 |
| Methanol | 75 | 198-200 | Brown | 4 |
| Benzene | 40 | 221-223 | White | 5 |
| Ethanol | 45 | 190-193 | Yellowish white | 6 |
| Ethanol | 40 | 204-206 | Yellow | 7 |
| DMF | 70 | 180-183 | Yellow | 8 |
| Ethanol | 86 | 110-112 | Brown | 9 |
| Ethanol | 76 | 225-228 | Reddish orange | 10 |

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